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AFML-TR-74-150

CORROSION CONTROL AT GRAPHITE/EPOXY – ALUMINUM AND TITANIUM INTERFACES

D. G. Treadway

General Dynamics Convair Division
San Diego, California

TECHNICAL REPORT AFML-TR-74-150
Final Report for Period January 1974 – June 1974

July 1974

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This report was supported by Director's Fund.

This technical report has been reviewed and is approved for publication.

Daniel E. Prince

DANIEL E. PRINCE
Project Monitor

FOR THE COMMANDER

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A test program was conducted to develop and evaluate corrosion protection systems for use on graphite/epoxy-aluminum and graphite/epoxy-titanium joints. The joint specimens were prepared in duplicate and protected with several corrosion protection systems including epoxy polyamide primer (MIL-P-23377C), inhibited polysulfide sealant, and a linear polyurethane topcoat.

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20. ABSTRACT.

All specimens were subjected to cyclic tension loading at -65°F to induce typical topcoat cracks around fasteners followed by one week of 100% humidity at 120°F and four weeks of exfoliation salt spray at 120°F. Visual observations were made and joint strength degradation determined.

It was concluded that graphite/epoxy-aluminum and titanium interface areas demanded careful corrosion protection, but conventional materials and techniques were adequate. The joint strength of the specimens tested were not degraded by exposure to a corrosive environment, with the exception of one adhesively bonded specimen.

FOREWORD

This report was prepared by the Convair Division of General Dynamics, San Diego, California under Air Force Contract F33615-74-C-5052. It was initiated under Project No. 7340, "Nonmetallic and Composite Materials," Task No. 734007, "Coatings for Energy Utilization, Control and Protective Functions." The work was administered under the direction of the Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio 45433. The contract was administered under the technical direction of the Elastomers and Coatings Branch, Nonmetallic Materials Division, with Mr. D. E. Prince (AFML/MBE) as project engineer.

This report covers the work carried out during the period from 11 January 1974 through 10 May 1974.

This report was submitted by the authors in June 1974.

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SUMMARY

Graphite/epoxy composite materials exhibit electropotentials similar to noble metals. Joints between bare graphite/epoxy and aluminum alloys have been shown to promote corrosion of the aluminum. A test program was conducted to evaluate various corrosion protection systems for use on graphite/epoxy-aluminum and graphite/epoxy-titanium joints.

Forty-eight lapped plate joint specimens were made using Type AS-3501 graphite/epoxy and 2024-T81 aluminum alloy. The six fasteners per specimen were countersunk machine screws prepared from aluminum, cadmium-plated steel, and titanium. Similar joint specimens were bonded using 3M AF143 adhesive cured at 350°F. The joint specimens were prepared in duplicate and protected with six corrosion protection systems including epoxy polyamide primer (MIL-P-23377C), inhibited polysulfide sealant, and a linear polyurethane topcoat.

All specimens were subjected to cyclic tension loading at -65°F to induce typical topcoat cracks around fasteners. One of each pair of specimens was then subjected to one week of humidity at 120°F and four weeks of exfoliation salt spray at 120°F. Visual observations were made and joint strength degradation noted for each configuration. No strength degradation occurred because of corrosion and, in general, attack observed visually was slight. Corrosion was no worse than that noted on aluminum-aluminum control specimens when protected only with the standard organic coating system. However, one conventionally protected adhesive-bonded specimen did have interface attack, and a more sophisticated protection system is indicated.

Similarly, 24 lapped plate joint specimens were prepared using Type AS-3501 graphite/epoxy and Ti-6Al-4V alloy. The six fasteners per specimen consisted of countersunk machine screws prepared from titanium and CRES A286. The 3M AF143 adhesive system was also used. Four protection systems were tested. Exposure and testing were identical to that described for the graphite/epoxy-aluminum joint specimens, and again no joint degradation occurred. The only visual corrosion consisted of significant red rust corrosion of the CRES A286 fasteners. Even coated CRES fasteners were attacked if cracks were present. No structural damage was apparent on the CRES fasteners, but staining of the specimen was extensive. No attack of the titanium fasteners or the adhesive system was noted.

It is concluded that graphite/epoxy-aluminum and titanium interface areas demand careful corrosion protection, but conventional sealing and surface coating materials and techniques are adequate. The element test data accumulated indicates that future work should be done with more complex structures at the component level.

SECTION 1

INTRODUCTION

Graphite/epoxy material technology, with its strength-to-weight and stiffness advantages, has advanced to the point where it promises considerable impact on aerospace structures. Certain shortcomings, however, are evident that must be further identified and evaluated. One is possible corrosive effects to fasteners and adjacent structure fabricated of other materials.

Graphite/epoxy composites can ordinarily be considered highly inert, corrosion-resistant materials. This is true when they are joined to themselves or to other inert materials. Unfortunately, when joined to many structural metals, they act like highly noble metals and promote corrosion of the less noble metals. When coupled with aluminum, for example, the potential difference can be more than one volt – enough driving force to cause considerable corrosion of the anodic aluminum.

The most critical area of an aircraft exterior finish system is around fastener areas. These areas are precisely where graphite/epoxy might be in contact with dissimilar metals in the form of structural members and fasteners. Past experience has shown that finish systems for these areas must be exceptionally good to prevent serious corrosion when conventional aircraft materials are used (Reference 1). The performance of these current protection systems with graphite/epoxy, however, may not be good enough due to the noble-metal characteristics of graphite/epoxy.

Several approaches could offer better corrosion protection. These include insulation, protection from moisture, use of corrosion inhibitors, graded galvanic protection, and reduction of cathode area. The objective of this investigation was to evaluate corrosion protection systems representing several of these approaches along with the conventional protection system to determine the magnitude of the graphite/epoxy metal interface problem. If the problem proved formidable, the study was to recommend possible directions for future work.

SECTION 2

SPECIMEN DESIGN

2.1 STRUCTURAL

The objective of this test program was to determine environmental effects on the strength of joints connecting graphite/epoxy composite material to metal structure. The joints are, therefore, sized to be critical at the interface between the composite and the metal. In the case of mechanical attachments, the joints are sized to be critical in bearing and fastener shear. For bonded joints, the joints are to be critical in bond shear.

Due to the wide scatter in bearing strengths of graphite/epoxy, bearing stresses are calculated simply as $f_{br} = P/Dt$, which is consistent with the bearing design strengths reported in the Design Guide for Advanced Composites (Reference 2). Fastener single shear strengths are as reported in MIL-HDBK-5B. Shear distribution among the fasteners is assumed uniform. Specimen geometry and materials are shown in Table 1.

The three fasteners for the graphite/epoxy-aluminum panels are aluminum, cadmium-plated steel, and titanium. They are reasonable candidates for the graphite/epoxy-aluminum joints since they are all galvanically compatible with the aluminum and with protection may be good with the graphite/epoxy. Aluminum and cadmium-plated steel are inexpensive and, therefore, it would be beneficial to know if they could be used with an adequate corrosion protection system. Titanium would be the best fastener selection since it is close to graphite/epoxy galvanically and is cathodic to aluminum. It also has a good strength to weight ratio.

The two fasteners for the graphite/epoxy-titanium joints were titanium and CRES A286. Because of the relatively high strength of titanium and graphite/epoxy, high-strength fasteners are indicated. Aluminum fasteners have too low a shear strength to be used efficiently and are also anodic to both titanium and graphite/epoxy. Therefore, as a fastener, aluminum would be highly subject to galvanic attack, accentuated by an unfavorable anode-to-cathode ratio. Cadmium-plated steel is also unsuitable because the cadmium causes embrittlement of titanium alloys at elevated temperatures. Titanium fasteners are excellent because they have a noble electropotential, in the same region as graphite/epoxy, and they have a good strength-to-weight ratio. Another compatible fastener would be CRES A286 – an iron base alloy.

The fasteners are flush, 100-degree head machine screws. The flush head was selected as typical of exterior construction where exposure is most severe. A small (No. 6) diameter was selected as being most probable of showing measurable reductions in

Aluminum	1,2	3,4	5,6	7,8	9,10	11,12	13,14	15,16	17,
Test Material Configuration Number	1	1	1	1	1	1	2	2	2
Fixture Material Configuration Number	5	5	5	5	5	5	6	6	6
Test Material (See Note 1)	G	G	G	G	G	G	G	G	G
Fixture Material (See Note 1)	A	A	A	A	A	A	A	A	A
Test Material Dimensions (See Note 2)	B	B	B	B	B	B	F	F	F
Fixture Material Dimensions (See Note 3)	C	C	C	C	C	C	G	G	G
Fastener Material (See Note 4)	A	A	A	A	A	A	B	B	B
Protection System No.	2	3	4	5	6	7	2	3	4
Protection System Type (See Note 5)	A	B	C	D	E	F	A	B	C

Titanium	49,50	51,52	53,54	55,56	57,58	59,60	61,62	63,64	65,
Test Material Configuration Number	17	17	17	17	18	18	18	18	[illegible]
Fixture Material Configuration Number	20	20	20	20	21	21	21	21	[illegible]
Test Material (See Note 1)	G	G	G	G	G	G	G	G	[illegible]
Fixture Material (See Note 1)	T	T	T	T	T	T	T	T	[illegible]
Test Material Dimensions (See Note 2)	E	E	E	E	E	E	E	E	[illegible]
Fixture Material Dimensions (See Note 3)	F	F	F	F	F	F	F	F	[illegible]
Fastener Material (See Note 4)	D	D	D	D	C	C	C	C	[illegible]
Protection System No.	9	10	11	12	9	10	11	12	[illegible]
Protection System Type (See Note 5)	H	B	A	A	H	B	A	A	[illegible]

NOTES:

1. G = AS/3501 graphite/epoxy
   A = 2024-T81 aluminum alloy
   T = Ti-6Al-4V titanium alloy

2. A = 3 × 6 × 0.040
   B = 3 × 6 × 0.060
   C = 3 × 6 × 0.080
   D = 3 × 6 × 0.100
   E = 3 × 6 × 0.120
   F = 3 × 6 × 0.150

3. A = 3 × 6.56 × 0.080
   B = 3 × 7.17 × 0.120
   C = 3 × 7.27 × 0.120
   D = 3 × 7.17 × 0.160
   E = 3 × 7.17 × 0.200
   F = 3 × 7.27 × 0.240
   G = 3 × 7.27 × 0.300

4. A = [illegible]
   B = [illegible]
   C = [illegible]
   D = [illegible]
   E = [illegible]

Specimen No.																						
,4	5,6	7,8	9,10	11,12	13,14	15,16	17,18	19,20	21,22	23,24	25,26	27,28	29,30	31,32	33,34	35,36	37,38	39,40	41,42	43,44	45,46	47,48
1	1	1	1	1	2	2	2	2	2	2	3	3	3	3	3	3	4	4	4	4	4	4
5	5	5	5	5	6	6	6	6	6	6	7	7	7	7	7	7	8	8	8	8	8	8
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A
B	B	B	B	B	F	F	F	F	F	F	E	E	E	E	E	E	A	A	A	A	A	A
C	C	C	C	C	G	G	G	G	G	G	F	F	F	F	F	F	A	A	A	A	A	A
A	A	A	A	A	B	B	B	B	B	B	D	D	D	D	D	D	E	E	E	E	E	E
3	4	5	6	7	2	3	4	5	6	7	2	3	4	5	6	7	2	3	4	5	6	7
B	C	D	E	F	A	B	C	D	E	F	A	B	C	D	E	F	A	B	C	D	E	F

Specimen No.																	
,52	53,54	55,56	57,58	59,60	61,62	63,64	65,66	67,68	69,70	71,72	73,74	75,76	77,78	79,80	81,82	83,84	85,86
17	17	17	18	18	18	18	19	19	19	19	9	10	11	12	23	24	25
20	20	20	21	21	21	21	22	22	22	22	13	14	15	16	26	27	28
G	G	G	G	G	G	G	G	G	G	G	A	A	A	A	T	T	T
T	T	T	T	T	T	T	T	T	T	T	A	A	A	A	T	T	T
E	E	E	E	E	E	E	A	A	A	A	B	D	C	A	B	B	A
F	F	F	F	F	F	F	A	A	A	A	B	E	D	A	B	B	A
D	D	D	C	C	C	C	E	E	E	E	A	B	D	E	D	C	E
10	11	12	9	10	11	12	9	10	11	12	1	1	1	1	8	8	8
B	A	A	H	B	A	A	H	B	A	A	A	A	A	A	G	G	G

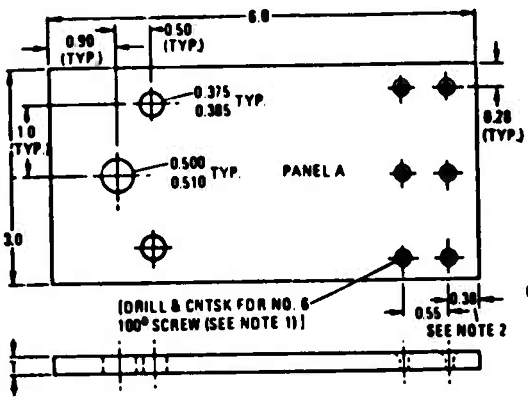
3. A = 3 × 6.56 × 0.080
   B = 3 × 7.17 × 0.120
   C = 3 × 7.27 × 0.120
   D = 3 × 7.17 × 0.160
   E = 3 × 7.17 × 0.200
   F = 3 × 7.27 × 0.240
   G = 3 × 7.27 × 0.300

4. A = Aluminum alloy (AN507DD)
   B = Cadmium plated steel (NAS1151-6)
   C = Cres A286 (NAS1151-E4)
   D = Titanium alloy (NAS1151-V4)
   E = 350° F adhesive (3M AF-143)

5. A = Insulative inhibited coating
   B = Insulative plastic barrier film
   C = Insulative inhibited sealant
   D = Galvanic control metallic film
   E = Galvanic control electroless plating
   F = Insulative anodic coating
   G = Uncoated; sealant on fasteners/joint
   H = Uncoated; inhibitive primer on bonded joint

Table 1. Joint Specimen Geometry and Materials

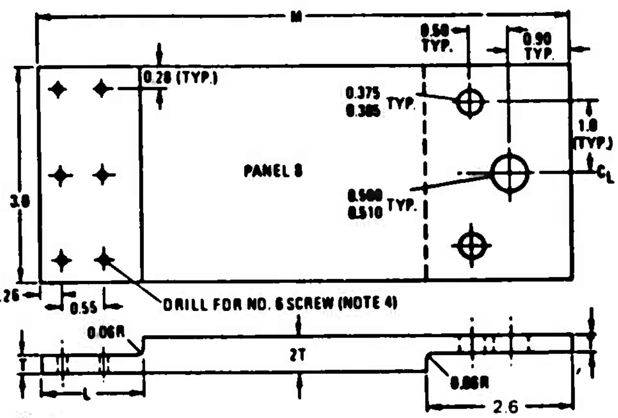
43,44	45,46	47,48
4	4	4
8	8	8
G	G	G
A	A	A
A	A	A
A	A	A
E	E	E
5	6	7
D	E	F



NOTE:
1. FASTENER HOLES NOT REQUIRED FOR CONFIG. 4, 12, 16, & 25
2. CHANGE 0.38 TO 0.26 FOR 2024-T81 & Ti-6Al-4V

MATERIAL: AS/3501 G/E $[0/\pm45]$ FOR 0.040 = $[0_2/\pm45]_s$
EXCEPT AS SPECIFIED

T (IN.)	0.060	0.150	0.120	0.040	0.040
CONFIG.	1	2	3, 17, 18	4	19
MATERIAL = 2024-T81 Al ALLOY					
T	0.060	0.100	0.080	0.040	
CONFIG.	6	10	11	12	
MATERIAL = Ti-6Al-4V					
T	0.060	0.060	0.040		
CONFIG.	23	24	25		



NOTE:
3. DIMENSIONS IN INCHES
4. FASTENER HOLES NOT REQUIRED FOR CONFIG. 8, 16, 22 & 28

MATERIAL = 2024-T81 ALUMINUM ALLOY								
T	0.080	0.150	0.120	0.040	0.060	0.100	0.080	0.040
M	7.27	7.27	7.27	6.56	7.17	7.17	7.17	6.56
CONFIG.	5	6	7	8	13	14	15	16
L	1.30	1.30	1.30	0.56	1.20	1.20	1.20	0.59
MATERIAL = Ti-6Al-4V								
T	0.120	0.120	0.040	0.060	0.080	0.040		
M	7.27	7.27	6.56	7.17	7.17	6.56		
CONFIG.	20	21	22	26	27	28		
L	1.30	1.30	0.59	1.20	1.20	0.56		

film
film
s plating
ners/joint
r on bonded joint

3

strength due to corrosion. An added benefit of the small diameter is that it is possible to develop the shear strength of the higher-strength fasteners with the fairly low bearing strength graphite/epoxy. The fasteners are listed in Table 2.

The specimens were subjected to stress analysis to determine critical sections, to predict ultimate strengths, and to set fatigue loading levels. All specimens were checked for fastener shear, fastener bearing ultimate, fastener bearing yield, net section tension, and fixture eccentricity.

The fatigue spectrum considered was represented by two loading blocks comprising 160 cycles at a peak load level of 85 percent of the predicted ultimate static strength and 50,000 cycles at a peak load level of 45 percent of predicted ultimate. The peak test loads used for each specimen configuration are shown in Table 3. Bearing yield strength was found to be critical for the small diameter countersink bolts used in some joints. Additionally, some of the test fixtures were found to be critical. Derivations of ultimate strength and test load levels for one graphite/epoxy-aluminum joint are presented as an example in the following discussion.

Table 2. Fasteners

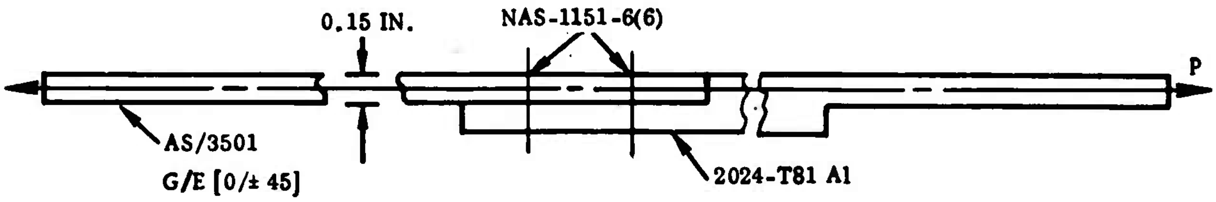
	Material			
	2024 Al	Alloy Steel	CRES A-286	Ti-6Al-4V
Machine Screws				
Finish	Anodized	Cad. Plated	Passivated	None
Part No.	AN507DD632R8*	NAS1151-6	NAS 1151E4	NAS 1151V4
Head Height (in.)	0.060/0.051	0.057 ref.	0.057 ref.	0.057 ref.
Grip (in.)	0.125	0.375 ±0.010	0.250 ±0.010	0.250 ±0.010
Length (in.)	$0.500\ ^{+0.000}_{-0.020}$	0.651 ±0.015	0.526 ±0.015	0.526 ±0.015
F_{su} (KS)	38	108	95	95
Single Shear Strength (lb)	568	1615	1421	1421
Nuts				
Finish	Cad. Plated	Cad. Plated	Silver Plated	Cad. Plated
Part No.	MS21042L06	MS21042L06	MS21043-06	MS21042L06
Height (in.)	0.115/0.141	0.115/0.141	0.115/0.141	0.115/0.141
Max. Dia. Base (in.)	0.244	0.244	0.244	0.244

* Except 0.125 grip from top of head to start thread.

Table 3. Cyclic Test Loads for Joint Specimens

Test Material	Fixture Material	Fastener Material	Load for 160 cycles (lb)	Load for 50000 cycles (lb)
graphite/epoxy	aluminum	aluminum	1850	980
	aluminum	steel	5860	3100
	aluminum	titanium	4680	2480
	aluminum	adhesive	1280	680
	titanium	titanium	5130	2710
	titanium	stainless steel	5130	2710
	titanium	adhesive	1510	800
aluminum	aluminum	aluminum	1850	980
	aluminum	steel	3690	2100
	aluminum	titanium	3120	1650
	aluminum	adhesive	1280	680
titanium	titanium	titanium	4290	2270
	titanium	stainless steel	4290	2270
	titanium	adhesive	1510	800

The ultimate static strength analysis considered a graphite/epoxy-aluminum specimen with steel fasteners.



Single shear strength of NAS 1151 bolt = 1421 lb (Ref. MIL-HDBK-5B)

Shear strength of bolts = 1421 x 6 = 8526 lb

Bearing strength in graphite/epoxy is:

Bearing strength of NAS 1151 bolts in [0/±45] graphite/epoxy

$$P_{BRU} = F_{BRU}\, D t n$$

F_{BRU} [0/±45] layup = 77000 psi (Ref. Design Guide, Figure 2.4.2-31)

$$P_{BRU} = 77000 \times 0.138 \times 0.15 \times 6 = 9563 \text{ lb}$$

Tension on net section of the graphite/epoxy at loaded holes is:

$$P \text{ (allowable)} = \frac{F_{TU}}{K_T} (W - 3D)\, t$$

K_T = 3.0 (Ref. Design Guide Figure 2.4.2-7)

F_{TU} 0±45 graphite/epoxy = 70000 psi

$$P \text{ (allowable)} = \frac{70000}{3.0} (3.0 - 3 \times 0.14)\ 0.15 = 9030 \text{ lb}$$

Tension of the graphite/epoxy at net section at grips is:

$$P \text{ (allowable)} = \frac{F_{TU}}{K_T} (W - 2D)\, t = \frac{70000}{3.0} (3.0 - 2 \times 0.375)\ 0.15 = 7875 \text{ lb}$$

Eccentric axial load on aluminum net section at the joint is computed by assuming some end fixity at the grips, and a reduced eccentricity (65%):

$$0.65 \times 0.15 = 0.098 \text{ inch}$$

Axial tension stress on net section is then:

$$f_t = \frac{P}{(W - 3D)\, t} = \frac{P}{(3.0 - 3 \times 0.14)\ 0.15} = 2.58\ P$$

and bending stress on the net section is:

$$f_b = \frac{6\,(P\,e)}{(W - 3\,D)\, t^2} = \frac{6 \times 0.098\ P}{(3.0 - 3 \times 0.14)\ 0.15^2} = 10.13\ P$$

At failure:

$$\frac{f_t}{F_{TU}} + \frac{f_b}{F_B} = 1.0$$

where:

F_{TU} (2024-T81) = 68,000 psi

$F_B = 1.4\ F_{TU}$

Therefore:

$$\frac{2.58\,P}{68,000} + \frac{10.85\,P}{1.4 \times 68,000} = 1.0$$

$$P_{(allowable)} = \frac{1.0}{\frac{1}{68,000}\left[2.58 + \frac{10.13}{1.4}\right]} = 6900 \text{ lb}$$

The bearing yield of fasteners in graphite/epoxy was not reasonably available. Consequently, it was decided to use data in MIL-HDBK-5B as a first approximation. Using Table 8.1.5.2(a) and extrapolating down from diameters of 0.250 and 0.190 to a diameter of 0.138 and interpolating between thicknesses of 0.125 and 0.160 yields:

$$P_{bry} = 1101 \text{ lb/fastener } (t = 0.150,\ D = 0.138)$$

$$P_{yield} = 6 \times 1101 = 6606 \text{ lb}$$

In summary, allowable loads are as summarized in Table 4. The eccentrically loaded aluminum portion of the specimen is critical at 6900 pounds. For high stress load cycles:

$$P_{max} = 0.85\ P_{ULT} = 5860 \text{ lb}$$

Since this is less than bearing yield, it is satisfactory.

For low stress load cycles:

$$P_{max} = 0.45\ P_{ult} = 3100 \text{ lb}$$

Table 4. Graphite/Epoxy Allowable Loads

Failure Mode	Allowable Load (P_{ult})
Bolt Shear	8526
Bolt Bearing in graphite/epoxy	9563
Graphite/epoxy Tension on Net Section at Joint	9030
Graphite/epoxy Tension on Net Section at Grips	7875
Aluminum Eccentrical Axial Load at Joint	6900
Bolt Bearing Yield in Graphite/epoxy	6608

Adhesive-bonded specimens are included to evaluate the effects of corrosion due to environment on joint strength. Consequently, the specimens are sized to provide failure in the joint and not in net tension. A 0.5-inch overlap has been selected as typical of most test specimens.

Significant scatter exists in the fatigue life of adhesive-bonded joints. To ensure no failures during fatigue cycling, a low shear allowable of 1000 psi was assumed for the graphite/epoxy to aluminum joints. The graphite/epoxy to titanium joints have less thermal stress, and a higher allowable of 1180 was selected. The overlap area is 1.5 in^2. This gives conservative ultimate strengths of 1500 and 1770 pounds, respectively.

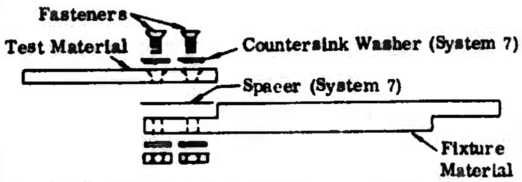
2.2 PROTECTION SYSTEMS

Corrosion-protection systems were selected on the basis of a representative system for each of several general concepts. These concepts were taken from corrosion theory and include: 1) insulation, 2) moisture exclusion, 3) polarization with corrosion inhibitors, 4) low cathode area, and 5) graded galvanic joints. Tables 5 and 6 and the following discussion describe the protection systems evaluated.

2.2.1 CORROSION PROTECTION SYSTEM NO. 1. This system was used for the aluminum-aluminum couple and was used as the control to compare joints with graphite/epoxy-aluminum couples. It represented one of the best current protection systems for aluminum structures. It used chemical film (MIL-C-5541B), inhibited epoxy primer (MIL-P-23377C), and a typical linear polyurethane topcoat. Fasteners were primed with epoxy primer and applied wet with unthinned epoxy primer. Bonded specimens were bonded to the bare solvent cleaned (MEK) substrate using a corrosion-inhibited adhesive primer followed by chemical film, epoxy primer, and topcoat. Butt joints and fastener heads were sealed with corrosion-inhibited polysulfide sealant, followed by the polyurethane topcoat. This system depended on a combination of insulation, inhibition, and moisture exclusion.

2.2.2 CORROSION PROTECTION SYSTEM NO. 2. This system was for graphite/epoxy-aluminum couples and represented a simple system very similar to System 1. Corrosion-inhibited polysulfide was used to seal the butt joints and fastener heads. This system depended on insulation to control the corrosion. The bonded joints are bare substrates primed with corrosion-inhibited primer. Sealant is used only at butt joint and fastener heads. If successful, this system would be one of the simplest solutions.

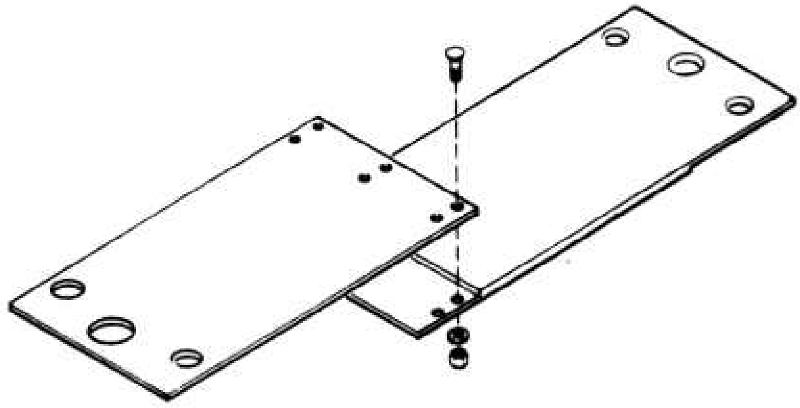
2.2.3 CORROSION PROTECTION SYSTEM NO. 3. System 3 was used for graphite/epoxy-aluminum couples and made use of an integrally bonded sheet of epoxy fiber cloth and resin (DuPont Kevlar 49) on each side of the composite. This should provide an insulator to protect the aluminum from the graphite. Discs of Kevlar 49 were



Protection System	Specimen Numbers	Fasteners				Configuration: Test Material			Configuration: Fixture Material		Assembly Processing	
		Aluminum (Anodized)	Steel (Cadmium Plated)	Titanium	CRES (Passivated)	Graphite/ Epoxy	Aluminum	Titanium	Aluminum	Titanium		Test Material
1	73,74 75,76 77,78	●	 ●	 ●			● (Control)		●		Install fasteners wet with epoxy polyamide primer (MIL-C-23377C). Polysulfide sealant (Products Research PR 1422G) in butt joint and around fastener heads. Epoxy polyamide primer (MIL-C-23377C) on washers and nuts. Polyurethane topcoat (DeSoto 821-T209) on entire assembly.	Chemical conversion coating (Alodine 1200S, MIL-C-5541B). Epoxy polyamide primer (MIL-C-23377C).
2	1,2 13,14 25,26	●	 ●	 ●		●			●		Same as System 1.	Epoxy polyamide primer (MIL-C-23377C).
3	3,4 15,16 27,28	●	 ●	 ●		●			●		Same as System 1 except install fasteners using Kevlar 49 countersink washers.	Interface side has Kevlar 49 as final laminate. Use epoxy polyamide primer (MIL-C-23377C).
4	5,6 17,18 29,30	●	 ●	 ●		●			●		Same as System 1 except: Install fasteners wet with PR 1422G sealant instead of with epoxy polyamide primer (MIL-C-23377C). Apply polysulfide faying surface sealant (Products Research PR 1431G) to interface area.	Epoxy polyamide primer (MIL-C-23377C).
5	7,8 19,20 31,32	●	 ●	 ●		●			●		Same as System 1.	Bond 4-mil 1100-H18 aluminum foil to interface area using Hysol EA 934. Epoxy polyamide primer (MIL-C-23377C).
6	9,10 21,22 33,34	●	 ●	 ●		●			●		Same as System 1.	Apply copper/electroless nickel on both sides of interface area. Epoxy polyamide primer (MIL-C-23377C).
7	11,12 23,24 35,36	●	 ●	 ●		●			●		Same as System 1 except also: Place an aluminum alloy 2024-T3 clad spacer between the panels. The spacer is processed with hard anodize and epoxy polyamide primer (MIL-C-23377C). Install fasteners wet with epoxy polyamide primer (MIL-P-23377C) and use a hard anodized countersink washer.	Epoxy polyamide primer (MIL-C-23377C).
8	81,82 83,84			●	 ●			● (Control)		●	High-temperature sealant (Teledyne Pro-seal 899B) in butt joint and around fastener heads. Epoxy polyamide primer (MIL-C-23377C) on washers and nuts.	None
9	49,50 57,58			●	 ●	o				●	Same as System 8.	None
10	51,52 59,60			●	 ●	●				●	Same as System 8 except install fasteners using Kevlar 49 countersink washers.	Interface side has Kevlar 49 as final laminate.
11	53,54 61,62			●	 ●	●				●	Same as System 8 except also: Install fasteners wet with epoxy polyamide primer (MIL-C-23377C) Use polyurethane topcoat (DeSoto 821-T209) on test material panel only.	Epoxy polyamide primer (MIL-C-23377C).
12	55,56 63,64			o	 ●	●				●	Same as System 8 except also: Install fasteners wet with epoxy polyamide primer (MIL-C-23377C). Use polyurethane topcoat (DeSoto 821-T209) on both panels (test material and fixture material).	Epoxy polyamide primer (MIL-C-23377C).

Table 5. Protection Systems Used for Specimens with Machine-Screw Fasteners

Detail Processing		
Test Material	**Fastener**	**Fixture Material**
Chemical conversion coating (Alodine 1200S, MIL-C-5541B). Epoxy polyamide primer (MIL-C-23377C).	Epoxy polyamide primer (MIL-C-23377C).	Chemical conversion coating (Alodine 1200S, MIL-C-5541B). Epoxy polyamide primer (MIL-C-23377C).
Epoxy polyamide primer (MIL-C-23377C).	Same as System 1.	Same as System 1.
Interface side has Kevlar 49 as final laminate. Use epoxy polyamide primer (MIL-C-23377C).	Same as System 1.	Same as System 1.
Epoxy polyamide primer (MIL-C-23377C).	Same as System 1.	Same as System 1.
Bond 4-mil 1100-H18 aluminum foil to interface area using Hysol EA 934. Epoxy polyamide primer (MIL-C-23377C).	Same as System 1.	Same as System 1.
Apply copper/electroless nickel on both sides of interface area. Epoxy polyamide primer (MIL-C-23377C).	Same as System 1.	Same as System 1.
Epoxy polyamide primer (MIL-C-23377C).	Same as System 1.	Same as System 1.
None	None	None
None	None	None
Interface side has Kevlar 49 as final laminate.	None	None
Epoxy polyamide primer (MIL-C-23377C).	Epoxy polyamide primer (MIL-C-23377C).	None
Epoxy polyamide primer (MIL-C-23377C).	Epoxy polyamide primer (MIL-C-23377C).	Epoxy polyamide primer (MIL-C-23377C).



Specimen No.	Protection System	Specimen No.	Protection System	Specimen No.	Protection System
1	2	21	6	54	11
2	2	22	6	55	12
3	3	23	7	56	12
4	3	24	7	57	9
5	4	25	2	58	9
6	4	26	2	59	10
7	5	27	3	60	10
8	5	28	3	61	11
9	6	29	4	62	11
10	6	30	4	63	12
11	7	31	5	64	12
12	7	32	5	73	1
13	2	33	6	74	1
14	2	34	6	75	1
15	3	35	7	76	1
16	3	36	7	77	1
17	4	49	9	78	1
18	4	50	9	81	8
19	5	51	10	82	8
20	5	52	10	83	8
		53	11	84	8

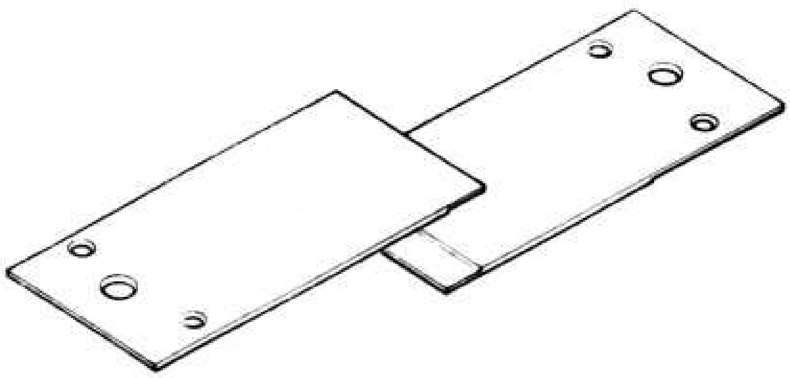
2

Table 6. Prot

Protection System	Specimen Numbers	Configuration: Test Material: Graphite/Epoxy	Test Material: Aluminum	Test Material: Titanium	Fixture Material: Aluminum	Fixture Material: Titanium	Assembly Processing (Test Material; Al Aly Foil (System 5) or Al Aly Spacer (System 7); Fixture Material)	Detail Processing: Test Material	Detail Processing: Fixture Material
1	79, 80		● (Control)		●		Adhesive bond using 3M Co. AF 143 adhesive. Chemical conversion coating touchup (Alodine 1200S) on aluminum portion. Epoxy-polyamide primer (MIL-P-23377C). Polysulfide sealant (Products Research PR 1422 G) in butt joint Polyurethane topcoat (DeSoto 821-T20 on entire assembly.	Adhesive primer (American Cyanamid BR 127) on bond interface area.	Adhesive primer (American Cyanamide BR 127) on bond interface area.
2	37, 38	●			●		Same as System 1.	None	Same as System 1.
3	39, 40	●			●		Same as System 1.	Interface side has Kevlar 49 as final laminate.	Same as System 1.
4	41, 42	●			●		Same as System 1.	None	Same as System 1.
5	43, 44	●			●		Same as System 1 except add a 4-mil aluminum alloy foil in the interface area using 3M Co. AF 143 adhesive.	None	Same as System 1.
6	45, 46	●			●		Same as System 1.	Apply copper/electroless nickel on both sides of interface area.	Same as System 1.
7	47, 48	●			●		Same as System 1 except also place a 2024-T3 clad aluminum alloy spacer between the panels. The spacer is processed with hard anodize. Bond with 3M Co. AF 143 adhesive.	None	Same as System 1.
8	85, 86			●	(Control)	●	Adhesive bond using 3M Co. AF 143 adhesive. High-temperature sealant (Teledyne Pro-seal 899B).	None	None
9	65, 66	●				●	Same as System 8.	None	None
10	67, 68	●				●	Same as System 8.	Interface side has Kevlar 49 as final laminate.	None
11	69, 70	●				●	Same as System 8 except also use: Epoxy polyamide primer (MIL-C-23377G) on test material panel only. Polyurethane topcoat (DeSoto 821-T209) on test material panel only.	None	None
12	71, 72	●				●	Same as System 8 except also use: Epoxy polyamide primer (MIL-P-23377C) on both panels. Polyurethane topcoat (DeSoto 821-T209) on entire assembly.	None	None

Table 6. Protection Systems Used for Adhesive-Bonded Specimens

	...al Processing
	Fixture Material
...eri-	Adhesive primer (American Cyanamide BR 127) on bond interface area.
	Same as System 1.
...lar	Same as System 1.
	Same as System 1.
	Same as System 1.
...ess ...f	Same as System 1.
	Same as System 1.
	None
	None
...lar	None
	None
	None



Specimen No.	Protection System	Specimen No.	Protection System
37	2	65	9
38	2	66	9
39	3	67	10
40	3	68	10
41	4	69	11
42	4	70	11
43	5	71	12
14	5	72	12
45	6	79	1
46	6	80	1
47	7	85	8
48	7	86	8

2

placed in the countersink areas to insulate the fastener heads. Details of these specimens were prepared and assembled like system No. 1, using chemical film, epoxy primer, inhibited sealant, and polyurethane topcoat. Bonded specimens had bare substrates and corrosion-inhibited adhesive primer. This system emphasized insulation as a means of controlling corrosion between dissimilar materials.

2.2.4 CORROSION PROTECTION SYSTEM NO. 4. This system was used for graphite/epoxy-aluminum couples and represented a system very similar to system No. 1, but with the addition of a corrosion-inhibited polysulfide sealant applied to the faying surfaces and to the fasteners as they were installed. It was also used to seal the butt joints and fastener heads. This system depended on insulation, corrosion inhibition, and polarizing agents to control corrosion. The bonded joints used bare substrates primed with corrosion-inhibited adhesive primer. Sealant was used only at the butt joint on bonded specimens.

2.2.5 CORROSION PROTECTION SYSTEM NO. 5. This system was based on bonding aluminum foil (4 mil) to the graphite/epoxy, thus creating an aluminum-aluminum "working" joint, which should respond to the same protection systems as for aluminum-aluminum joints. Details and fasteners were treated in the same manner as system No. 1. The bonded specimens were prepared similar to system No. 1. This system depended on changing the cathodic side of the joint to make it similar to the anodic side, thus reducing the potential difference.

2.2.6 CORROSION PROTECTION SYSTEM NO. 6. This system depended on placing an intermediate metal in the galvanic couple between the noble graphite and the anodic aluminum to reduce the overall corrosive effect. The graphite/epoxy was electroplated with copper (pyrophosphate) followed by electroless nickel plating. Since the nickel was still somewhat cathodic to the aluminum, the additional protection of system No. 1 was also applied. The bonded specimens were coated with corrosion-resistant adhesive primer before bonding.

2.2.7 CORROSION PROTECTION SYSTEM NO. 7. The final system for graphite/epoxy-aluminum consisted of a hard-anodized spacer that provided insulation and abrasion resistance in the joint. The hard anodized material consisted of 2024-T3 clad aluminum alloy processed to a thickness of 2 mils and sealed with 5% sodium dichromate solution at 200°F for 10 minutes. Anodized aluminum discs were used in each countersunk hole for the same purpose. All details, including the shims, received the System No. 1 coatings. Bonding was also the same as System No. 1. This system emphasizes insulation and abrasion resistance.

2.2.8 CORROSION PROTECTION SYSTEM NO. 8. This system, for titanium-titanium specimens, was the control for graphite/epoxy-titanium couple specimens. Since little corrosive action was expected between titanium and titanium, the control specimen was left bare. A high-temperature, corrosion-resistant sealant (Coast Proseal 899B) was used to fill the butt joint and fasteners.

2.2.9 CORROSION PROTECTION SYSTEM NO. 9. This system was for the first graphite/epoxy-titanium couple. Since little corrosive action was expected between graphite/epoxy and titanium due to little difference in the galvanic series, a minimum protection was expected to be adequate. It was identical to System No. 8.

2.2.10 CORROSION PROTECTION SYSTEM NO. 10. This system is similar to System No. 3, and uses the insulation technique. Epoxy cloth and resin (Kevlar 49) were integrally bonded to the graphite/epoxy. Kevlar 49 washers were used under the heads of all fasteners. Bonded specimens were adhered directly to the bare titanium.

2.2.11 CORROSION PROTECTION SYSTEM NO. 11. This system used finishes for protection. Only the graphite/epoxy was painted with epoxy primer and polyurethane topcoat. Fasteners were installed with unthinned epoxy primer. This system was based on restricting the area of the cathode, thus resulting in a large anode-to-cathode area ratio.

2.2.12 CORROSION PROTECTION SYSTEM NO. 12. This system assumed that the titanium and graphite/epoxy both require protection. It was the same as System No. 11, except that both the graphite/epoxy and titanium received epoxy primer and polyurethane topcoat. The system depended on a combination of insulation, inhibition, and moisture exclusion.

SECTION 3

SPECIMEN PREPARATION

3.1 GRAPHITE/EPOXY FABRICATION

Approximately 16 pounds of AS/3501 prepreg were received from Hercules Incorporated. Prepreg properties were run and conformed to requirements as follows:

	Requirement	Actual
Fiber strength, ksi	390 (minimum)	414
Fiber modulus, msi	28 to 34	32.3
Resin content, %	39 to 45	42.1
Volatiles, %	3 (maximum)	0.92
Flow	15 to 25	15.1

The material had good drape and tack.

A total of eight graphite/epoxy panels were fabricated from this prepreg. Four of the panels were graphite/epoxy with thicknesses of 0.040, 0.060, 0.120, and 0.150 inch. The remaining four were identical, except for one ply of Kevlar 49, Style 181, on one side. All panels consisted of 0- and ±45-degree plies laid up symmetrically. A total of 72 graphite/epoxy half-specimens were machined from these eight panels.

The fabrication procedure for the eight panels was:

a. Prepare a clean aluminum base plate for layup.

b. Put down one layer of Teflon film.

c. Stack graphite/epoxy plies in the desired orientation.

d. Cover with a layer of Armalon separator.

e. Place bleeder system in position (1-ply Style 120 cloth for every 4 plies of prepreg).

f. One layer Teflon film.

g. Vent system (2 plies 181, 1 ply 1534 cloth).

h. Vacuum bag.

i. Cure. (Apply a minimum of 27 in. Hg vacuum. Heat at 2 to 3°F/minute to 350°F. Apply 100 psi autoclave pressure as the temperature reaches 225°F. Hold at temperature for one hour. Cool below 150°F before removing pressure.)

3.2 MACHINING

Machining of the aluminum and titanium materials was accomplished with high speed steel cutters on a standard milling machine. Industry-recommended speeds and feeds were used throughout the sawing, milling, drilling, and countersinking operation.

The graphite/epoxy material was sawed to length using a special Convair large diamond disc composite cutoff saw. The saw blade used is a 10-inch diameter, 46-grit diamond impregnated by 0.050-inch-thick and run at a cutting speed of 5600 sfm and a feed rate of 8 inches per minute. A water-soluble oil was was used as a coolant.

All holes in the graphite/epoxy material were drilled on a vertical mill, with cobalt high-speed-steel twist drills at a drilling speed of 50 sfm. The feed rate was hand fed, and no coolant was used. Countersinking operations were also performed on the milling machine using a diamond-plated 60/80 grit countersink at a drilling speed of 1500 sfm.

3.3 PROCESSING

Processing details and sequences for all joint specimens are given in Tables 7 through 9.

Processing Steps								
	1,2	3,4	5,6	7,8	9,10	11,12	13,14	1[illegible]
Identify using metal stamps (specimen number).	●	●	●	●	●	●	●	
Hand wipe with aliphatic naphtha.	●	●	●	●	●	●	●	
Age harden 2024-T3 aluminum alloy to 2024-T81 by heating to 365-385° F for 12 ±1/2 hours.	●	●	●	●	●	●	●	
Clean panels using methyl ethyl ketone.								
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.								
Apply Alodine 1200S chemical film per MOS1-02674 (MIL-C-5541B).	●	●	●	●	●	●	●	
Clean bond interface on titanium panel using HNO_3-HF per MOS1-02801-008B.								
Clean bond interface on aluminum panel using FPL solution.								
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.	●	●	●	●	●	●	●	
Apply 0.2-0.4 ml of BR127 adhesive primer to the bond interface areas.								

SPECIMEN NUMBER																								
Fasteners																Bonded						F		
5,6	7,8	9,10	11,12	13,14	15,16	17,18	19,20	21,22	23,24	25,26	27,28	29,30	31,32	33,34	35,36	37,38	39,40	41,42	43,44	45,46	47,48	49,50	51,52	53,54
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•			
																						•	•	•
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•									
																•	•	•	•	•	•			
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•									
																•	•	•	•	•	•			

Table 7. Processing of Test Fixture Panels

...IMEN NUMBER																						
...onded				Fasteners								Bonded				Fasteners			Bonded	Fasteners		Bonded
...,42	43,44	45,46	47,48	49,50	51,52	53,54	55,56	57,58	59,60	61,62	63,64	65,66	67,68	69,70	71,72	73,74	75,76	77,78	79,80	81,82	83,84	85,86
●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●
●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●
●	●	●	●													●	●	●	●			
				●	●	●	●	●	●	●	●	●	●	●	●					●	●	●
							●				●											
																●	●	●				
												●	●	●	●							●
●	●	●	●																●			
																●	●	●				
●	●	●	●																			

3

Table 8. Processing of Test
Epoxy-Aluminum A

Processing Steps	Specimen N												
	1,2	3,4	5,6	7,8	9,10	11,12	13,14	15,16	17,18	19,20	21,22	23,24	25,
Identify by suitable means (specimen number).	•	•	•	•	•	•	•	•	•	•	•	•	•
Clean panels by hand wiping with methyl ethyl ketone.	•	•	•	•	•	•	•	•	•	•	•	•	•
Apply copper electroplating (pyrophosphate type) and electroless nickel plating to the graphite/epoxy-aluminum interface area (W by 1.5 in.length).					•						•		
Clean bond interface (rough side) on graphite/epoxy panel using Scotchbrite and MEK.				•						•			
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.	•	•	•		•	•	•	•	•		•	•	•
Place a hard anodized aluminum panel in the interface area between the graphite/epoxy and aluminum. Install hard anodized (W by 1.5 in. length) aluminum washers under each fastener. First coat (panels only) with epoxy primer except for Specimens 47 & 48.						•						•	
Bond 4 mil 1100-H18 aluminum foil to the graphite/epoxy faying surface using EA934 (W by 1.5 in. length, rough side), Hysol EA934 (0-00096-58).				•						•			
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.				•						•			
Clean fasteners by washing in clean aliphatic naphtha. Allow to dry. Handle with clean white cotton gloves.	•	•	•	•	•	•	•	•	•	•	•	•	•
Coat fasteners (head and shanks) with epoxy primer to apply a thin coating. Blot off excess on head. Air dry 1 hour. Accelerate cure by exposure to 150°F for 24 hours.	•	•	•	•	•	•	•	•	•	•	•	•	•
Wear clean white cotton gloves. Place a layer of 3M AF143 tape so that a 1/2-inch overlap exists on the two panels for the joint specimen. Cure at 35 psi and 350°F for 60 minutes.													
Apply 0.2-0.4 ml of BR127 adhesive primer to the bond interface areas.													
Bond 4 mil 1100-H18 aluminum foil to the graphite/epoxy faying surface using EA934 (W by 1.5 in. length, rough side), Hysol EA934 (0-00096-58).													
Wear clean white cotton gloves. Place a layer of 3M AF143 tape so that a 1/2-inch overlap exists on the two panels for the joint specimen. Cure at 35 psi and 350°F for 60 minutes.													
Dip fasteners (head and shanks) in unthinned epoxy primer. Blot off excess that collects on head. Install fasteners and wipe off excess.	•	•		•	•	•	•	•		•	•	•	•
Install titanium alloy screws (NAS1151-V4) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.													•
Dip fasteners in PR1422G sealant. After installation, wipe off excess. Air dry 1 hour. Heat to 130°F for 3 hours to accelerate cure.			•						•				
Prepare washers from Kevlar 49 and place under fasteners prior to installation.		•						•					
Apply PR1431G to faying surface of joint specimen. After installation of fasteners, wipe off excess. Cure 24 hours at 75°F followed by 48 hours at 130°F.			•						•				
Install titanium alloy screws (NAS1151-V4) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.													
Apply touch-up chromate conversion coating to aluminum only per MOS1-02674.													
Install aluminum alloy screws (AN507DD) by holding the head stationary and tightening the nut with a torque wrench to a value of 1.8-2.5 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.	•	•	•	•	•	•							
Install cadmium-plated steel screws (NAS1151-6) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.							•	•	•	•	•	•	
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.													
Fill butt joint gap with PR1422G fillet sealant. Air dry 1 hour. Heat to 130°F for 3 hours to accelerate cure.	•	•	•	•	•	•	•	•	•	•	•	•	
Fill fastener heads with PR1422G fillet sealant. Air dry 1 hour. Heat to 130°F for 3 hours to accelerate cure.	•	•	•	•	•	•	•	•	•	•	•	•	
Spray-apply one uniform wet-tack coat of topcoat. Allow to dry for 10 minutes and follow with two uniform wet cross-coats to a combined total topcoat dry film thickness of 4 mils. Allow first wet cross-coat to dry 30 to 40 minutes before applying second wet cross-coat. After 2-hour air dry (minimum), heat to 140°F for 24 hours.	•	•	•	•	•	•	•	•	•	•	•	•	

Table 8. **Processing of Test Material Panels and Graphite/ Epoxy-Aluminum Assembled Specimens**

Specimen No.																						
3,4	5,6	7,8	9,10	11,12	13,14	15,16	17,18	19,20	21,22	23,24	25,26	27,28	29,30	31,32	33,34	35,36	37,38	39,40	41,42	43,44	45,46	47,48
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
			•						•						•						•	
		•						•						•			•	•	•	•	•	•
•	•		•	•	•	•	•		•	•	•	•	•		•	•						
				•						•						•						•
		•						•						•								
		•						•						•								
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•						
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•						
																	•	•	•		•	•
																						•
																				•		
																						•
•		•	•	•	•	•		•	•	•	•	•		•	•	•						
											•			•	•	•						
	•						•						•									
•						•						•										
	•						•						•									
												•	•									
																	•	•	•	•	•	•
•	•	•	•	•																		
					•	•	•	•	•	•												
																	•	•	•	•	•	•
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•						
•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•

2

Table 9. Processing of Test Material Panels and Graphite/Epoxy-Titanium Assembled Specimens

Processing Steps	Specimen No.																		
	49,50	51,52	53,54	55,56	57,58	59,60	61,62	63,64	65,66	67,68	69,70	71,72	73,74	75,76	77,78	79,80	81,82	83,84	85,86
Identify by suitable means (specimen number).	•	•	•	•	•	•	•	•	•	•	•	•							
Identify using metal stamps (specimen number).													•	•	•	•	•	•	•
Hand wipe with aliphatic naphtha.													•	•	•	•	•	•	•
Clean panels by hand wiping with methyl ethyl ketone.	•	•	•	•	•	•	•	•	•	•	•	•							
Clean bond interface (rough side) on graphite/epoxy panel using Scotchbrite and MEK.									•	•	•	•							
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.			•	•			•	•											
Age harden 2024-T3 aluminum alloy to 2024-T81 by heating to 365-385°F for 12 ±1/2 hours.													•	•	•	•			
Clean panels using methyl ethyl ketone.																	•	•	•
Clean fasteners by washing in clean aliphatic naphtha. Allow to dry. Handle with clean white cotton gloves.	•	•	•	•	•	•	•	•									•	•	
Coat fasteners (head and shanks) with epoxy primer to apply a thin coating. Blot off excess on head. Air dry 1 hour. Accelerate cure by exposure to 150°F for 24 hours.			•	•			•	•											
Wear clean white cotton gloves. Place a layer of 3M AF143 tape so that a 1/2-inch overlap exists on the two panels for the joint specimen. Cure at 35 psi and 350°F for 60 minutes.									•	•	•	•							
Dip fasteners (head and shanks) in unthinned epoxy primer. Blot off excess that collects on head. Install fasteners and wipe off excess.			•	•			•	•											
Install titanium alloy screws (NAS1151-V4) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.			•	•															
Install CRES A286 screws (NAS 1151-E4) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max)							•	•											
Apply Alodine 1200S chemical film per MOS1-02674 (MIL-C-5541B).													•	•	•				
Clean bond interface on titanium panel using HNO_3-HF per MOS1-02801-008B.																			•
Clean bond interface on aluminum panel using FPL solution.																•			
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.													•	•	•				
Apply 0.2-0.4 ml of BR127 adhesive primer to the bond interface areas.																•			
Clean fasteners by washing in clean aliphatic naphtha. Allow to dry. Handle with clean white cotton gloves.													•	•	•				
Coat fasteners (head and shanks) in epoxy primer to apply a thin coating. Blot off excess on head. Air dry 1 hour. Accelerate cure by exposure to 150°F for 24 hours.													•	•	•				
Dip fasteners (head and shanks) in unthinned epoxy primer. Blot off excess that collects on head. Install fasteners and wipe off excess.													•	•	•				
Install titanium alloy screws (NAS1151-V4) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.															•				
Wear clean white cotton gloves. Place a layer of 3M AF143 tape so that a 1/2-inch																•			•

value (PT). PT = 10 in.-lb, max)													•	•	•				
Apply Alodine 1200S chemical film per MOS1-02674 (MIL-C-5541B).																			•
Clean bond interface on titanium panel using HNO_3-HF per MOS1-02801-008B.																•			
Clean bond interface on aluminum panel using FPL solution.													•	•	•				
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.																•			
Apply 0.2-0.4 ml of BR127 adhesive primer to the bond interface areas.													•	•	•				
Clean fasteners by washing in clean aliphatic naphtha. Allow to dry. Handle with clean white cotton gloves.													•	•	•				
Coat fasteners (head and shanks) in epoxy primer to apply a thin coating. Blot off excess on head. Air dry 1 hour. Accelerate cure by exposure to 150°F for 24 hours.													•	•	•				
Dip fasteners (head and shanks) in unthinned epoxy primer. Blot off excess that collects on head. Install fasteners and wipe off excess.															•				
Install titanium alloy screws (NAS1151-V4) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.																•			•
Wear clean white cotton gloves. Place a layer of 3M AF143 tape so that a 1/2-inch overlap exists on the two panels fc. the joint specimen. Cure at 35 psi and 350°F for 60 minutes.																•			
Apply touch-up chromate conversion coating to aluminum only per MOS1-02674.																•			
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.			•	•			•	•	•	•	•	•							•
Fill butt joint gap with high-temperature sealant (FMS 1043). Air dry 72 hours. (Accelerated cure: room temperature for 4 hours, then heat to 140°F until cured.)													•						
Install aluminum alloy screws (AN507DD) by holding the head stationary and tightening the nut with a torque wrench to a value of 1.8-2.5 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.														•					
Install cadmium-plated steel screws (NAS1151-6) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.													•	•	•	•			
Fill butt joint gap with PR1422G fillet sealant. Air dry 1 hour. Heat to 130°F for 3 hours to accelerate cure.		•				•													
Prepare washers from Kevlar 49 and place under fasteners prior to installation.																	•		
Install titanium alloy screws (NAS1151-V4) by holding the head stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). (PT = 10 in.-lb, max.	•	•																	
Install CRES A286 screws (NAS1151-E4) by holding the heat stationary and tightening the nut with a torque wrench to a value of 9.1-11.4 in.-lb over the prevailing torque value (PT). PT = 10 in.-lb, max.					•	•												•	
Fill butt joint gap with high-temperature sealant (FMS1043). Air dry 72 hours. (Accelerated cure: room temperature for 4 hours, then heat to 140°F until cured.)	•	•			•	•											•	•	
Apply one uniform wet cross-coat of epoxy primer to a dry film thickness of 0.6-0.9 mil. Air dry one hour.												•							
Fill fastener heads with high-temperature sealant (FMS 1043). Air dry 72 hours. (Accelerated cure: room temperature for 4 hours, then heat to 140°F until cured.)	•	•	•	•	•	•	•	•									•	•	
Apply epoxy primer to test material only.											•								
Apply topcoat to test material only.											•								
Fill fastener heads with PR1422G fillet sealant. Air dry 1 hour. Heat to 130°F for 3 hours to accelerate cure.													•	•	•				
Spray-apply one uniform wet-tack coat of topcoat. Allow to dry for 10 minutes and follow with two uniform wet cross-coats to a combined total topcoat dry film thickness of 4 mils. Allow first wet cross-coat to dry 30 to 40 minutes before applying second wet cross-coat. After 2-hour air dry (minimum), heat to 140°F for 24 hours.				•				•				•	•	•	•	•			
Apply topcoat to test material only			•				•												

SECTION 4

ENVIRONMENTAL EXPOSURES

4.1 CYCLIC TENSION LOADING

The joint specimens were subjected to cyclic tension-fatigue loading at -65° F (R = +0.1) in the facility shown in Figure 1. The loads used are discussed in Section 2.1.

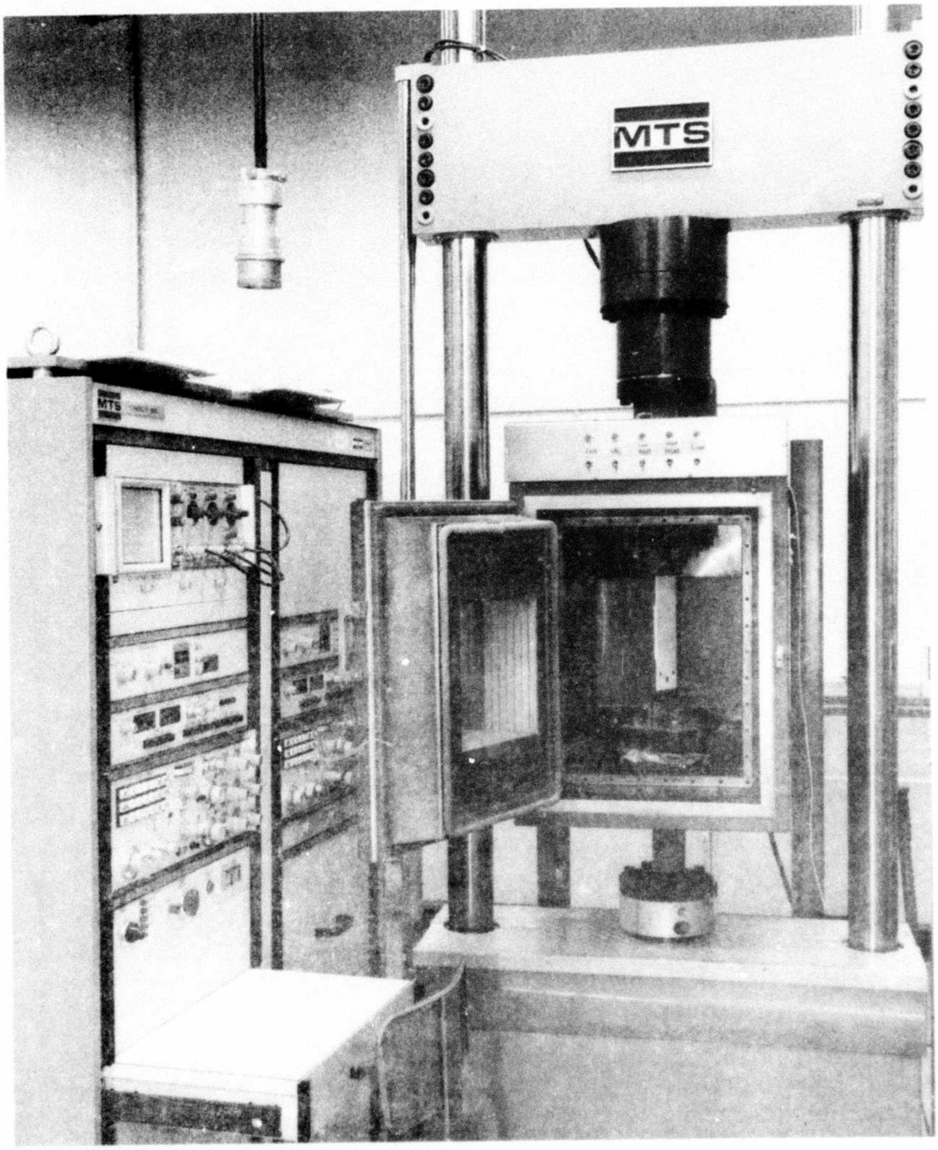


Figure 1. MTS Testing Machine with Cold Box and Typical Joint Specimen

4.2 HUMIDITY TESTING

The humidity test is described by Method 6201 of Federal Test Method Standard No. 141a. The cabinet is shown in Figure 2, and temperature was controlled to 120°F. This environment exposes the specimens to a moisture-saturated atmosphere at controlled temperature and with continuous condensation.

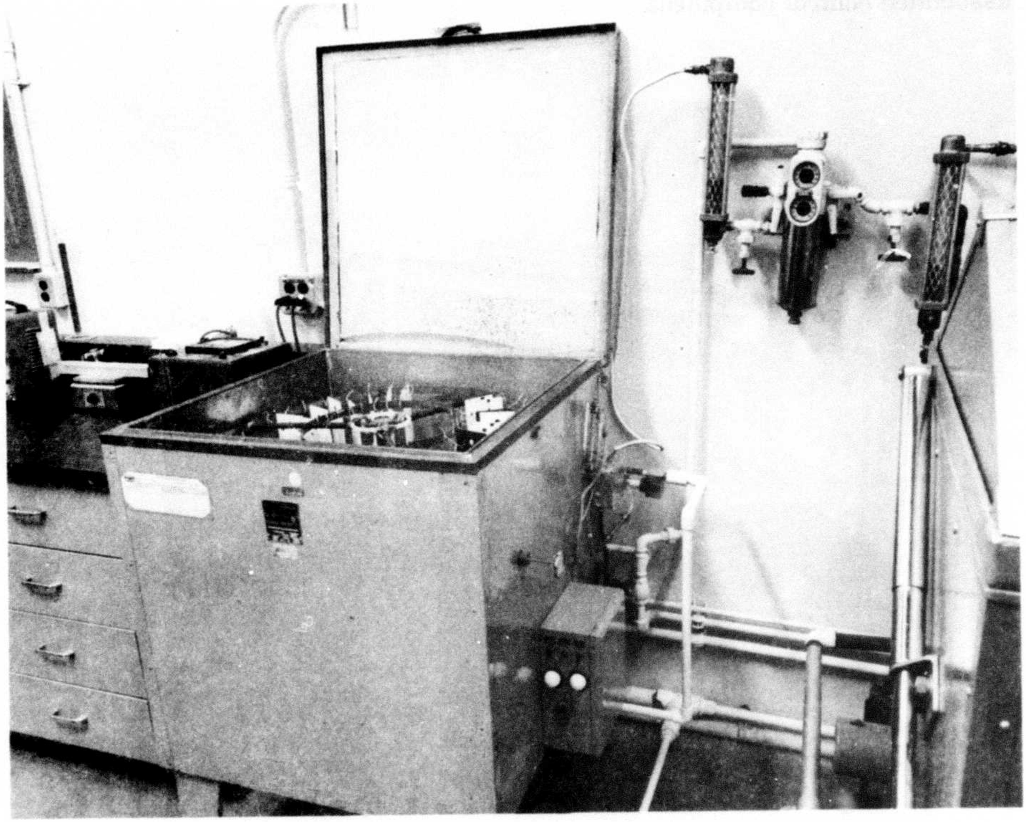


Figure 2. Humidity Cabinet

4.3 EXFOLIATION SALT SPRAY

The final accelerated corrosion test consisted of an exfoliation salt spray test. This test is similar to the standard 5 percent salt spray (ASTM B177) except it is cyclic. It alternately sprays the specimens, allows them to dry, and subjects them to high relative humidity. The salt solution is acidified with acetic acid to a pH of 3.0. Temperature is controlled at 120°F at all times. A typical cycle consisted of:

a. 45 minute spray.

b. 2-hour dry air purge.

c. 3 hour 15 minute soak at high relative humidity (achieved by using "wet bottom" condition in the chamber).

Each corrosion test specimen was exposed to this environment for 30 days. They were placed into the chamber at an angle of 6 degrees from the vertical. The test is an aggresive salt spray test, as described in Reference 3. Figure 3 shows the cabinet and associated control equipment.

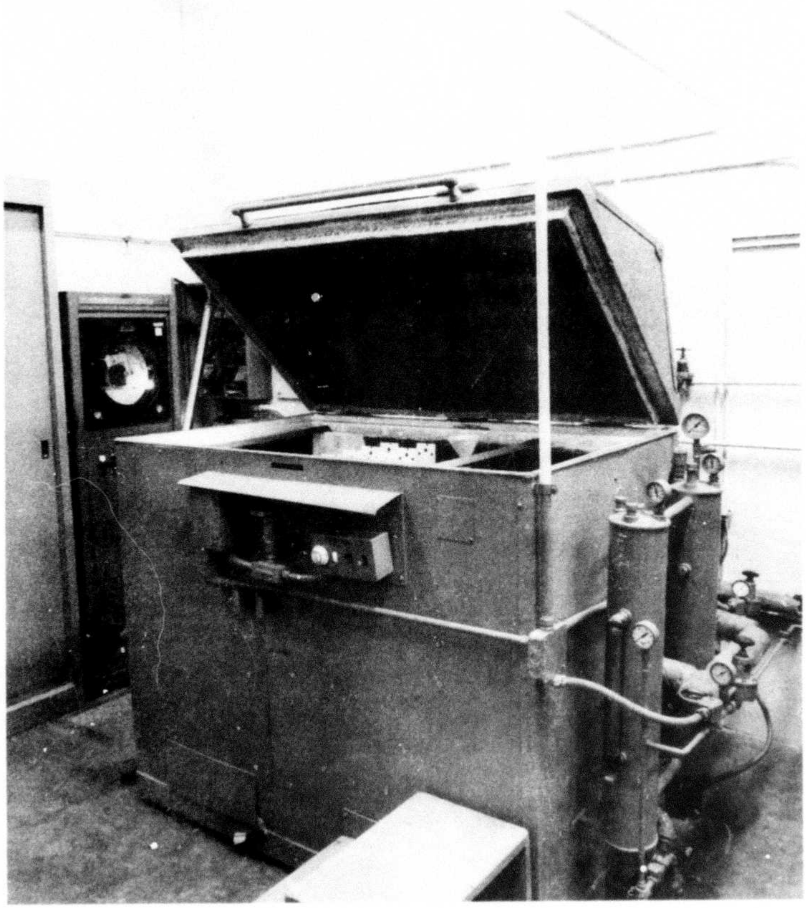


Figure 3. Exfoliation Salt Spray Cabinet

4.4 STATIC FAILURE LOADING

Each test specimen exposed to the corrosion test had an identical control specimen that was exposed to cyclic tension loading only. After tension loading, these control specimens were loaded to failure at -65°F. The corrosion test specimens were failed in an identical manner. Table 13 shows the failure loads for the specimens.

SECTION 5

FILM PROPERTY TESTS

5.1 MECHANICAL

One-half-inch tensile specimens of paints and sealants were prepared by casting on Teflon-coated plates. Ultimate tensile strength and total elongation were then determined for each material at -65°F (Table 10). These results show that the sealants are capable of about 10 times more elongation than paint films at -65°F. Loads used in cyclic testing of the specimens caused cracks at the fasteners in about 50 percent of the specimens, which means that local strain or the cyclic action of testing was sufficient to exceed the elongation capability of the paint. Since the underlying sealant had 10 times more capability for elongation, it probably did not crack. A paint that is more elastic at -65° F is needed to match the sealant.

Based on the low loss in joint strength and the good visual appearance of most specimens after exposure to the rigorous exfoliation salt spray environment, it appears that the mechanical properties of current state-of-the-art materials are adequate to provide corrosion protection of graphite/epoxy-aluminum and titanium joints.

Table 10. Mechanical Properties of Paints and Sealants at -65° F

Material	No.	Width (in.)	Thickness (in.)	Area (in^2)	Load (lb)	F_{tu} (lb)	Total Elongation (in./in.)
Paint Film Epoxy Primer (Mil-P-23377C) and Polyurethane Topcoat (DeSoto 821-T209)	1	0.500	0.0043	0.00215	13.4	6233	2.07
	2	0.500	0.0041	0.00205	14.3	6976	1.85
	3	0.500	0.0040	0.00202	12.1	6050	1.70
Polysulfide Sealant (Coast Proseal 899B) (Black)	1	0.500	0.0520	0.0260	32.0	1231	9.0
	2	0.500	0.0520	0.0260	37.5	1442	14.8
Polysulfide Sealant (Products Research 1422G) (Green)	1	0.517	0.0680	0.0352	46.1	1310	16.8
	2	0.497	0.0760	0.0378	39.0	1032	*
Polysulfide Sealant (Products Research 1431G) (Tan)	1	0.514	0.0600	0.0308	36.9	1198	16.2
	2	0.501	0.0820	0.0410	46.5	1134	20.0

* Broke inside doubler.

5.2 WATER PERMEABILITY

A method similar to ASTM D1653-62 was used to determine water permeability of three sealants and one paint film. Water permeability is defined as:

$$\frac{\text{(Water transmission per 24 hr, milligrams) (Thickness, mm)}}{\text{(Area, sq. cm.)}}$$

The results are listed in Table 11; weight gain curves are shown in Figure 4.

Water permeability of the sealants tested was all of the same order of magnitude. Specific permeability of the paint film is about 20 times superior to the sealants, indicating its value as a topcoat for sealants.

Since most protection systems tested included the paint film topcoat, there is little comparative data, but permeability of the paint film (0.086) was adequate to provide good protection for all systems. Yellow residues noted on some specimens indicated sufficient permeability of the sealant to permit migration of the chromate inhibitor.

Unpainted graphite/epoxy-titanium Specimens 57 and 59 showed no decrease in strength due to environmental tests, but did show some visual corrosion at CRES fasteners. Since this corrosion was not present on coated Specimens 53 and 55, the paint showed good moisture resistance.

Table 11. Water Permeability Results

Material Tested	Specific Permeability*
Polysulfide Sealant Coast Proseal 899B	1.5
Polysulfide Sealant Products Research 1422G	2.0
Polysulfide Sealant Products Research 1431G	4.6
Paint Film Epoxy Primer (Mil-P-23377C) Polyurethane Topcoat (DeSoto 821-T209)	0.086

*Milligrams of water transmitted per day through one square centimeter of a film one millimeter thick at room temperature when the relative humidity is 0% on one side and 100% on the other.

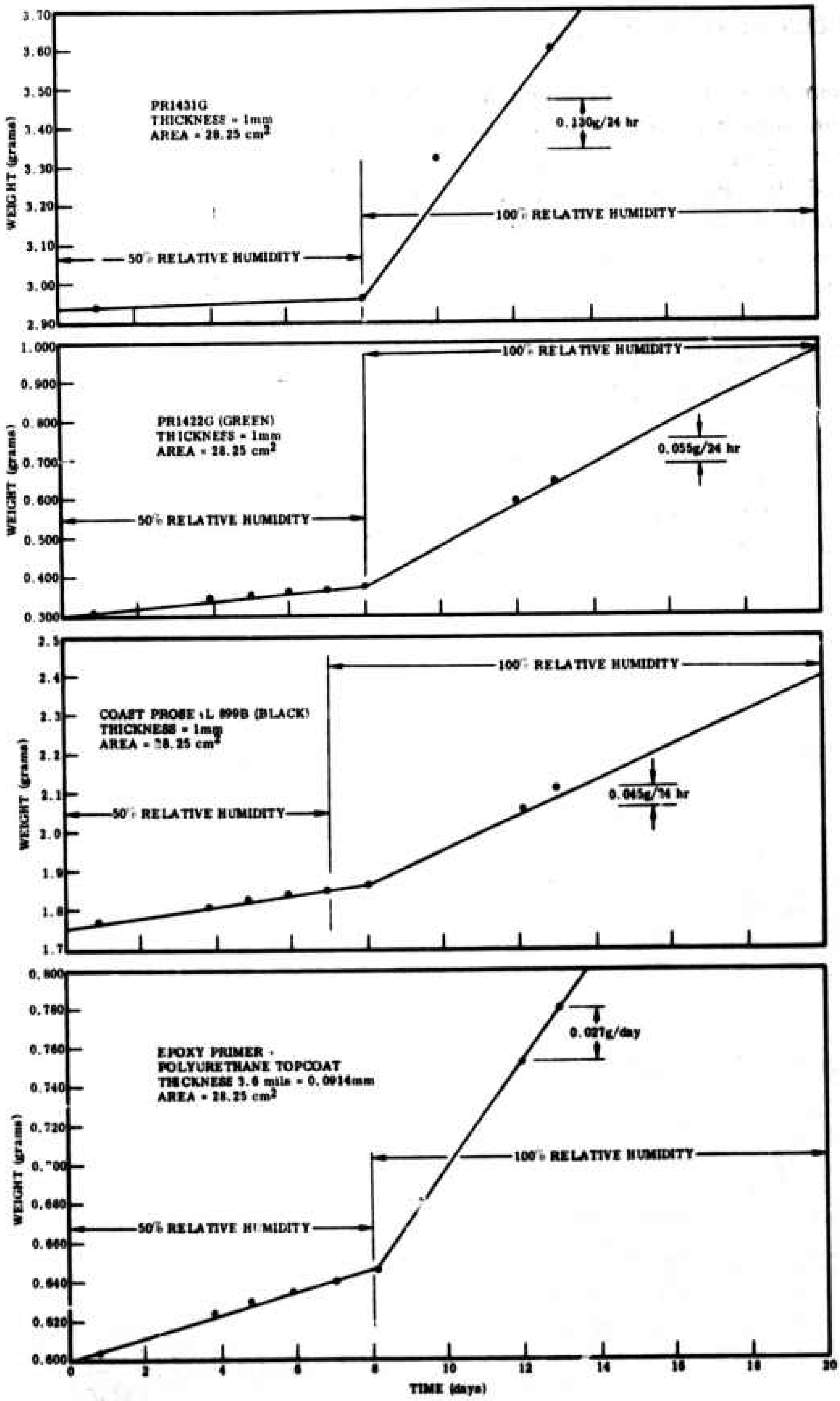


Figure 4. Water Transmission Curves

5.3 ELECTRICAL RESISTANCE

Measurements were made to determine if the degree of electrical insulation of the panel ends from each other and from the fasteners had any bearing on the degree of corrosion protection. Thirteen measurements for ohmic resistance were made for each specimen before and after each phase of the testing as shown in Figure 5. The first measurement is between the test material panel and the fixture material panel. The next six are between the test material panel and each of the fasteners and the next six between the fixture material panel and each of the fasteners. Contact with the fastener was made through a drop of saturated sodium chloride solution. Cracks in the coatings at the fasteners significantly influenced the resistance values. Resistance values for each specimen after each testing phase are listed in Table 12.

Ideally, there should be good correlation between high electrical resistance and good corrosion resistance. In the present tests, little correlation exists. This is due primarily to the fact that all protection systems were so adequate that the corrosive environment did not reach the area where the resistance was measured in the time

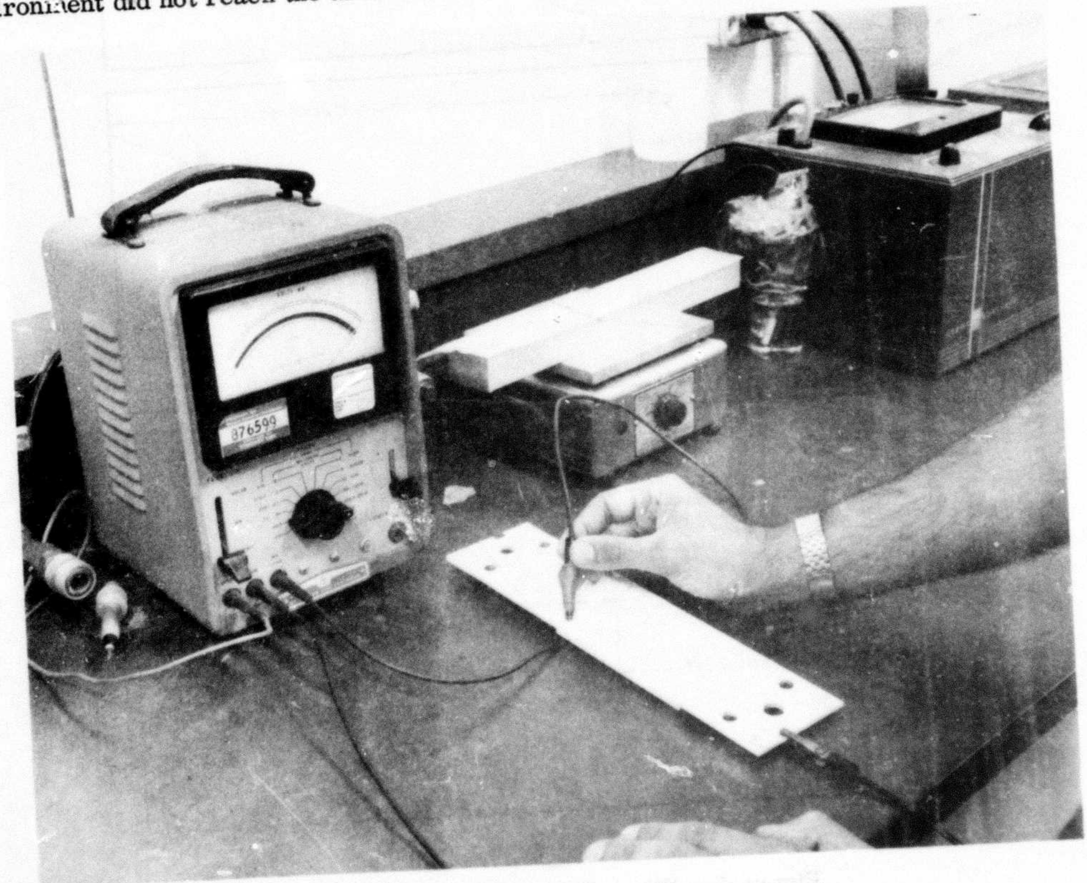


Figure 5. Electrical Resistance Test Set-Up

Resistance Path	1	3	5	7	9	12	13	15	17	19	21	23	25	27
From Graphite/Epoxy To Fastener No. 1	∞ 50000 90000	∞ 400000 100	∞ ∞ ∞	∞ ∞ ∞	∞ 1000 3.5	1000000 ∞ 1000	3000 200 40	∞ ∞ 100000	∞ ∞ 700000	15000 ∞ 50000	∞ ∞ 2.4	∞ ∞ 5000	∞ ∞ 5000000	∞ ∞ ∞
From Graphite/Epoxy To Fastener No. 2	∞ 40000 60000	∞ 300000 4000	∞ ∞ ∞	∞ ∞ ∞	∞ 10 1.5	∞ 100000 500	∞ 500 59	∞ ∞ ∞	∞ ∞ ∞	∞ ∞ 70000	∞ ∞ 70000	∞ ∞ ∞	∞ ∞ 50000	∞ ∞ ∞
From Graphite/Epoxy To Fastener No. 3	∞ 100000 50000	∞ 200000 300	∞ 90000 ∞	∞ ∞ ∞	500 30 10	∞ 40000 5000	∞ 1000 700	∞ ∞ ∞	∞ 200000 ∞	∞ ∞ ∞	∞ ∞ 50000	∞ ∞ ∞	∞ ∞ ∞	∞ 200000 25000
From Graphite/Epoxy To Fastener No. 4	∞ 5000 20000	5000 60000 100	∞ ∞ ∞	∞ ∞ ∞	150 8 1	∞ 50000 10000	18000 100 200	∞ ∞ ∞	∞ 3000000 200000	∞ ∞ ∞	∞ ∞ 30000	∞ ∞ ∞	∞ ∞ 40000	∞ ∞ ∞
From Graphite/Epoxy To Fastener No. 5	∞ 60000 20000	∞ 1000000 150	∞ 160000 ∞	∞ ∞ ∞	20 10 3	∞ 50000 400	∞ 300 200	∞ ∞ ∞	∞ 700000 ∞	∞ ∞ ∞	∞ ∞ 20000	∞ ∞ 1000000	∞ ∞ 100000	∞ ∞ ∞
From Graphite/Epoxy To Fastener No. 6	∞ 60000 20000	∞ 5000000 200	∞ ∞ ∞	∞ ∞ ∞	∞ ∞ 40	∞ ∞ 20000	∞ 2000 2000	∞ ∞ 70000	∞ 1000000 200000	∞ ∞ 200000	∞ 1000 20000	∞ ∞ 400000	∞ ∞ 10000	∞ 500000 35000
From Aluminum/ Titanium To Fastener No. 1	∞ 30000 40	∞ 400000 200	∞ ∞ ∞	∞ ∞ 1000	∞ 60000 5	1000000 70000 50	3000 400 50	∞ ∞ 150000	∞ ∞ 800000	∞ ∞ 200	∞ ∞ 3	∞ ∞ ∞	∞ ∞ 5000000	∞ ∞ ∞
From Aluminum/ Titanium To Fastener No. 2	∞ 30000 10	∞ 200000 2000	∞ 300000 5000000	∞ ∞ ∞	∞ 60000 3	∞ 70000 30	40000 300 20	∞ ∞ ∞	∞ ∞ ∞	∞ ∞ 100	∞ ∞ 13000	∞ ∞ ∞	∞ ∞ 100000	∞ ∞ ∞
From Aluminum/ Titanium To Fastener No. 3	∞ 70000 20	∞ 200000 100	∞ 100000 ∞	∞ ∞ ∞	35000 60000 5	∞ 80000 50	∞ 2000 3000	∞ ∞ ∞	∞ 200000 ∞	∞ ∞ ∞	∞ 3000 10	∞ ∞ ∞	∞ ∞ ∞	∞ 40000 160
From Aluminum/ Titanium To Fastener No. 4	∞ 30000 15	∞ 500000 150	∞ ∞ ∞	∞ ∞ ∞	40000 60000 2	∞ 60000 1000	∞ 400 170	∞ ∞ ∞	∞ ∞ 90000	∞ ∞ ∞	∞ ∞ 6	∞ ∞ 5000000	∞ ∞ 40000	∞ ∞ ∞
From Aluminum/ Titanium To Fastener No. 5	∞ 30000 200	∞ 200000 40	∞ 2000000 3000000	∞ ∞ ∞	40000 60000 5	∞ 60000 100	∞ 300 50	∞ ∞ ∞	∞ ∞ ∞	∞ ∞ ∞	∞ ∞ 10	∞ ∞ 800000	∞ ∞ 200000	∞ ∞ ∞
From Aluminum/ Titanium To Fastener No. 6	∞ 30000 20	∞ 5000000 45	∞ ∞ ∞	∞ ∞ ∞	∞ ∞ 3.5	∞ 500000 2400	∞ 2000 3000	∞ ∞ 18000	∞ 600000 200000	∞ ∞ 20000	∞ ∞ 5	∞ ∞ 300000	∞ ∞ 3000	∞ 500000 40000
From Aluminum/ Titanium To Graphite/ Epoxy	∞ 70000 16	∞ 300000 45	600000 30000 100000	130000 24000 500	40000 10000 0.600	∞ 150000 70	38000 200 30	19000 170000 120	1000000 37000 6000	45000 5000 200	500 2000 0.36	60000 200000 6000	0.18 0.018 0.024	∞ 130000 35

Note:

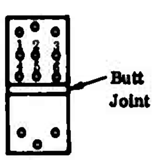
Three resistance measurements were made on each specimen. They are listed for each specimen in the following order:

xxxxxx After Cyclic
xxxxxx After Humidity
xxxxxx After 4-Week Exfoliation Salt Spray

A Hewlett-Packard 412A DC vacuum tube voltmeter and a saturated sodium chloride solution were used for all measurements.

	Specimen No.																		
	23	25	27	29	31	33	35	37	39	41	43	45	47	49	51	53	55	57	59
∞	∞	∞	∞	∞	∞	3000000	∞	Bonded	Bonded	Bonded	Bonded	Bonded	Bonded	40000	100000	∞	∞	200000	30000
∞	∞	∞	∞	1000000	∞	3000	∞							100000	90000	400000	∞	150000	50000
.4	5000	5000000	∞	40000	∞	200	150000							400	15	40000	∞	7	5
∞	∞	∞	∞	∞	∞	∞	∞							40000	100000	∞	∞	100000	40000
∞	∞	∞	∞	∞	∞	200	∞							100000	50000	∞	∞	200000	50000
00	∞	50000	∞	∞	∞	50	35000							130	20	∞	4400	10	5
∞	∞	∞	∞	∞	∞	∞	∞							40000	100000	∞	∞	500	50000
∞	∞	∞	200000	∞	∞	10	50000							500000	100000	50000	50000	150000	80000
00	∞	∞	25000	60000	∞	40	500							170	100	∞	200	7	3
∞	∞	∞	∞	∞	∞	∞	∞							40000	100000	∞	∞	70000	50000
∞	∞	∞	∞	∞	∞	18	∞							200000	50000	∞	∞	60000	50000
00	∞	40000	∞	400000	∞	10	50000							100	10	800000	8000	3	3
∞	∞	∞	∞	∞	∞	∞	∞							0.002	50000	∞	∞	100000	50000
∞	∞	∞	∞	∞	∞	300	40000							500000	80000	∞	∞	90000	50000
00	1000000	100000	∞	600000	∞	40	200000							500	50	∞	400	3	4
∞	∞	∞	∞	∞	∞	∞	∞							40000	50000	∞	∞	20000	50000
00	∞	∞	500000	400000	∞	80	50000							400000	50000	∞	∞	180000	50000
00	400000	10000	35000	30000	2800	40	20000							150	20	∞	300000	40	2
∞	∞	∞	∞	∞	∞	3000000								200	100	∞	∞	20000	30
∞	∞	∞	∞	1000000	∞	130	∞							4000	80	150000	400000	2000	60
3	∞	5000000	∞	100000	300000	50	50000							50000	30	2000	400000	10	20
∞	∞	∞	∞	∞	∞	∞	∞							20	20000	∞	∞	200	30
∞	∞	∞	∞	∞	∞	80	∞							300	150	∞	∞	3000	280
00	∞	100000	∞	∞	∞	50	30000							60000	30	∞	100000	20	16
∞	∞	∞	∞	∞	∞	∞	∞							2	200	∞	∞	50	1000
00	∞	∞	40000	∞	∞	6	70000							130000	300	1000	3000	10000	150
10	∞	∞	160	100000	300000	20	800							40000	50	∞	30000	10	18
∞	∞	∞	∞	∞	∞	∞	∞							30	200	∞	∞	300	50
∞	∞	∞	∞	∞	∞	10	0.05							2000	35	∞	∞	30	150
6	5000000	40000	∞	1000000	∞	20	20000							20000	50	∞	20000	10	16
∞	∞	∞	∞	∞	∞	∞	∞							40000	200	∞	∞	2000	200
∞	∞	∞	∞	∞	∞	20	∞							20000	1000	∞	100000	2000	400
10	800000	200000	∞	1000000	∞	35	200000							50000	100	∞	50000	10	16
∞	∞	∞	∞	∞	∞	∞	∞							50	200	∞	∞	20	50
∞	∞	∞	500000	260000	1000000	80	50000							1000000	40	∞	∞	100000	500
5	300000	3000	40000	40000	40000	40	8000							40000	50	∞	200000	20	16
500	60000	0.18	∞	∞	250000	260000	250000	0.3	∞	0.007	26000		∞	0.006	0.004	0.02	1000000	0.005	0.011
000	200000	0.018	130000	280000	1000	70000	160000	6	∞	0.007	400000		∞	0.005	0.008	0.05	30000	0.005	0.006
.36	6000	0.024	35	20000	1000	10	200	100000	30000	0.011	400000		200000	0.010	0.0007	0.10	200	70	0.015

oltmeter and a saturated
measurements.



2

Table 12. Resistance Measurements (k ohms)

51	53	55	57	59	61	63	65	67	69	71	73	75	77	79	81	83	85
100000	∞	∞	200000	30000	∞	∞	Bonded	Bonded	Bonded	Bonded	∞	∞	∞	Bonded	0.003	170000	Bonded
90000	400000	∞	150000	50000	∞	∞					1000	35000	∞		0.00006	40000	
15	40000	∞	7	5	240	∞					500	160000	200000		20	1.5	
100000	∞	∞	100000	40000	∞	∞					∞	∞	∞		20000	110000	
50000	∞	∞	200000	50000	∞	∞					90000	30000	∞		70000	50000	
20	∞	4400	10	5	50	∞					1000	240000	140000		15	1.2	
100000	∞	∞	500	50000	∞	∞					∞	∞	∞		100000	40000	
100000	50000	50000	150000	80000	∞	∞					60000	2000	70000		2000	10000	
100	∞	200	7	3	40	∞					5000	140000	100000		7	1.1	
100000	∞	∞	70000	50000	30000	∞					∞	∞	∞		100000	80000	
50000	∞	∞	60000	50000	∞	∞					6	4000	30000		4000	40000	
10	800000	8000	3	3	30	2000					1800	200000	70000		8	1.1	
50000	∞	∞	100000	50000	∞	∞					∞	∞	∞		100000	0.0001	
80000	∞	∞	90000	50000	∞	∞					1000	70000	1000000		1000	30000	
50	∞	400	3	4	1000000	∞					1000	110000	120000		10	1.1	
50000	∞	∞	20000	50000	∞	∞					∞	∞	∞		100000	500	
50000	∞	∞	180000	50000	1000000	∞					1000	300000	∞		5000	100000	
20	∞	300000	40	2	100000	∞					1000	110000	100000		14	1.1	
100	∞	∞	20000	30	∞	∞					∞	∞	∞		400	50000	
80	150000	400000	2000	60	∞	∞					3000	20000	∞		0.001	400	
30	2000	400000	10	20	500	∞					50	3	20000		30	20	
20000	∞	∞	200	30	400000	∞					∞	∞	∞		2000	30000	
150	∞	∞	3000	280	∞	∞					30000	20000	∞		18000	200	
30	∞	100000	20	16	100	∞					150	200	20000		20	10	
200	∞	∞	50	1000	∞	∞					∞	∞	∞		200	350	
300	1000	3000	10000	150	280	∞					50000	40000	400000		20	100	
50	∞	30000	10	16	50	∞					300	20	5000		10	15	
200	∞	∞	300	50	200	∞					∞	∞	∞		200	2400	
35	∞	∞	30	150	90	∞					160000	70000	260000		500	200	
50	∞	20000	10	16	50	30000					10	26	30000		10	10	
200	∞	∞	2000	200	∞	∞					∞	∞	∞		50000	10	
1000	∞	100000	2000	400	∞	∞					140000	170000	2000000		4000	50	
100	∞	50000	10	16	∞	∞					40	60	30000		20	5	
200	∞	∞	20	50	∞	∞					∞	∞	∞		50000	2000	
40	∞	∞	100000	500	200000	∞					120000	600000	2000000		1000	300	
50	∞	200000	20	16	∞	∞					35	3	20000		100	7	
0.004	0.02	1000000	0.005	0.011	0.010	200000	0.008	∞	∞	∞	26000	40000	70000	0.00016	0.0001	0.0001	0.0002
0.008	0.05	30000	0.005	0.006	0.006	0.600	0.008	4000000	1000000	140000	35000	100000	3000	0.001	0.00006	0.00006	0.0003
0.0007	0.10	200	70	0.015	0.008	40000	0.007	50000	100000	16000	100	13	800	0.011	0.0001	0.0001	0.0003

3

available. Another factor, as indicated by the large number of specimens that gained in strength, is that the normal variation in test data obscured the effect of the protection system.

The correlation between electrical resistance (as measured after the humidity exposure between test material and fixture material panels) and the change in joint strength after exfoliation salt spray exposure is shown in Figure 6.

Electrical resistance data from fasteners to end panels generally agreed well with test panel to fixture panel data. Most values were lowered as testing progressed, although the values increased in some cases. One explanation for this is the formation of higher resistance corrosion products.

Although little correlation was found to exist between electrical resistance (from test panel to fixture panel materials) and joint strength deterioration, there was good agreement between visual corrosion damage at specific fasteners and electrical resistance between that fastener and the panel ends. (See panel data for Specimens 3, 9, 33, and 73 in Tables 12 and 13. Panels 57, 59, and 61 showed red rust corrosion after the exfoliation test. Low resistance was not measured at these fasteners before the test, but it was appreciably lower after the test. On these panels, electrical resistance could not be used as a predictive test.

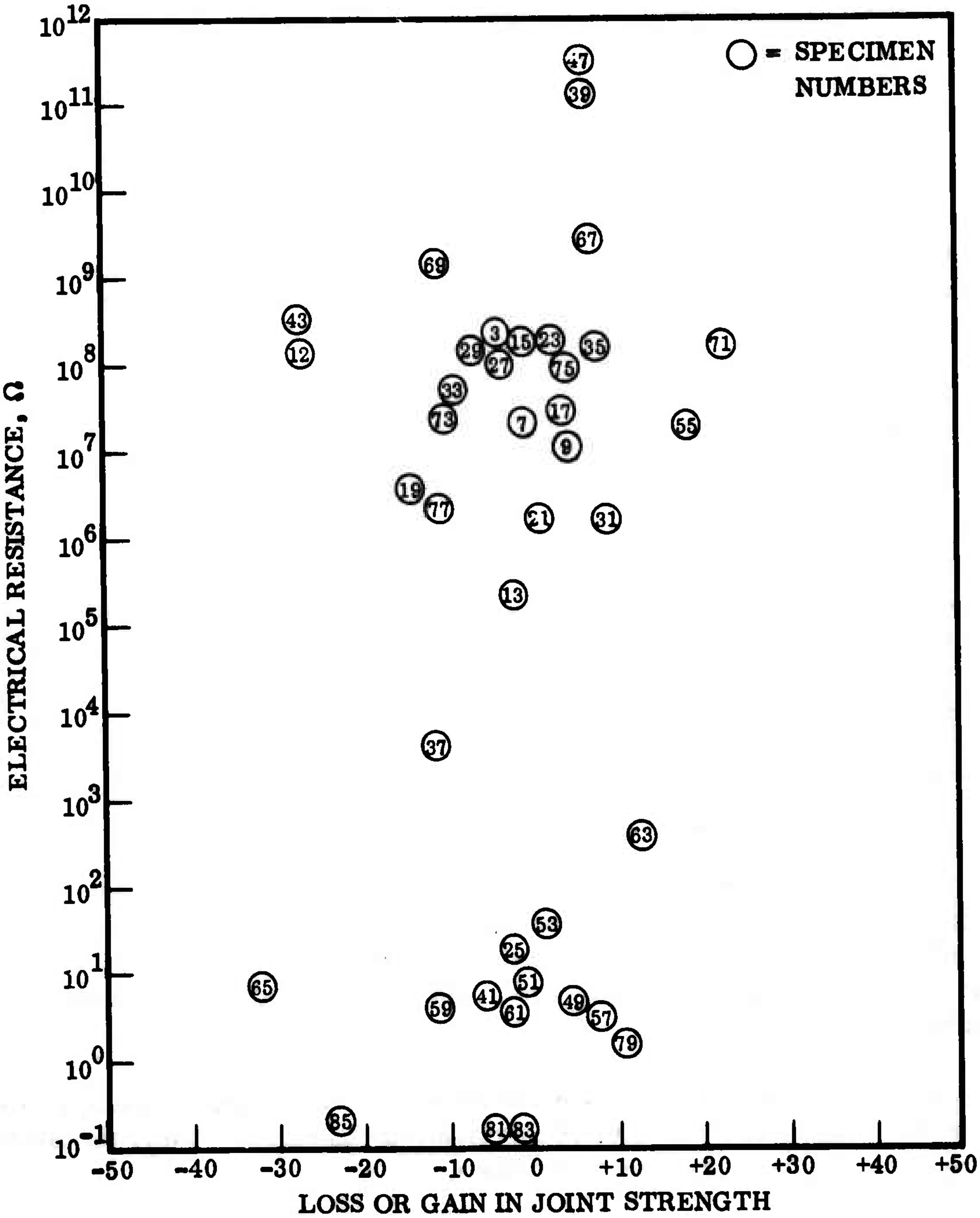


Figure 6. Comparison of Electrical Resistance with Change in Joint Strength

SECTION 6

SPECIMEN EVALUATION

6.1 VISUAL OBSERVATIONS

After a one-week exposure to the humidity test, each specimen was examined visually. No corrosion was noted. The only visual attack noted was blistering on some specimens.

A few blisters were noted on Specimens 3, 5, 15, 17, 25, 73, 75, and 77. Recovery occurred and the effect was insignificant. Blistering and adhesion loss were also noted on Specimens 9 and 21, but this was due to the copper/nickel plating. The titanium fixture panels on Specimens 55, 63, and 71 had significant blistering, but recovery occurred. Apparently, solvent cleaning of titanium is not adequate for optimum performance of the coating system.

Table 13 presents visual observations after the cyclic tension fatigue test, after two weeks of exfoliation salt spray, and after four weeks of exfoliation salt spray.

6.2 JOINT STRENGTH DEGRADATION

The joint strength of each control specimen and each corrosion specimen was determined at -65°F. The change in strength of the corrosion specimen was calculated and the failure mode of each specimen was noted. This information is presented in Table 13.

An examination of Table 13 reveals that the change in strength of the joint specimens was quite low in most cases, and no change in strength could be attributed to corrosion. The nature of fatigue testing accounts for some of the variability. In many cases of significant strength changes, the failure modes were such that these changes could be attributed to the mode itself. On Specimens 11 and 12, for example, the hard anodized spacer adhered to the graphite/epoxy so that the fasteners broke in a shear mode. This was not the case in the corrosion specimen. However, this did not happen because of corrosive degradation of the joint.

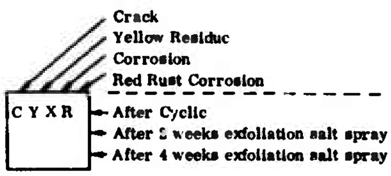
Some of the adhesive-bonded specimens had relatively large strength changes. A review, however, indicated an absence of corrosion, and in several cases, the nature of the bond break explained the results obtained.

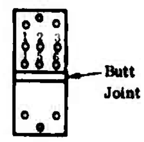
One shortcoming of this test program was the lack of duplicate test specimens. The strength data, therefore, must be correlated with visual observations.

Area Examined	1	3	5	7	9	12	13	15	17	19	21	23	25	27	29
Fastener 1	C - - - C Y - - C Y - -	C - - - C Y - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - - - C - - -	C - - - C - - - C - - -	C - - - C Y - - C Y - -	C - - - C - - - C - - -	- - - - - - - - - - - -	C - - - C Y - - C Y - -	C - - - C - - - C - - R	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - - - C - - -	- - - - - - - - - - - -
Fastener 2	C - - - C Y - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - X - C - X -	C - - - C - - - C - - -	C - - - C Y - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C Y - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -
Fastener 3	C - - - C Y - - C Y - -	C - - - C Y - - C - X -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - - - C - X -	C - - - C - - - C - - -	C - - - C Y - - C - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C Y - - C Y - -	C - - - C - - - C - - R	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - - - C - - -	- - - - - - - - - - - -
Fastener 4	C - - - C Y - - C - X -	C - - - C Y - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - X - C - X -	C - - - C - - - C - - -	C - - - C Y - - C - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - - R C - - R	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -
Fastener 5	C - - - C Y - - C Y - -	C - - - C Y - - C - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - - - C - X -	C - - - C - - - C - - -	C - - - C Y - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -
Fastener 6	C - - - C Y - - C Y - -	C - - - C Y - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - X -	- - - - - - - - - - - -	C - - - C Y - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - - R C - - R	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -
Butt Joint, Front	C - - - C Y - - C Y - -	C - - - C Y - - C Y - -	- - - - - - - - - Y - -	- - - - - - - - - - - -	- - - - - - - - - - X -	C - - - C Y - - C Y - -	C - - - C Y - - C Y - -	- - - - - Y - - - Y - -	C - - - C Y - - C Y - -	C - - - C - - - C - - -	C - - - C - - - C - X -	C - - - C Y - - C - - -	C - - - C Y - - C Y - -	C - - - C - - - C Y - -	C - - - C - - - C Y - -
Butt Joint, Side	- - - - - Y - - - - - -	C - - - C - - - C Y - -	- - - - - - - - - - - -	- - - - - - - - - Y - -	- - - - - - - - - - - -	C - - - C - - - C - - -	C - - - C - - - C Y - -	- - - - - - - - - Y - -	C - - - C - - - C - - -	C - - - C - - - C Y X -	C - - - C - - - C - X -	C - - - C - - - C - X -	C - - - C - - - C - X -	C - - - C - - - C Y - -	- - - - - - - - - - - -
Butt Joint, Back	C - - - C Y - - C Y - -	C - - - C - - - C - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -	C - - - C - - - C - X -	C - - - C Y - - C Y - R	- - - - - - - - - Y - R	C - - - C Y - - C Y - -	C - - - C Y - - C Y - -	C - - - C - - - C Y X -	- - - - - - - - - - - R	- - - - - - - - - - - R	C - - - C - - - C Y - -	- - - - - - - - - - - R

FAILURE DATA	1	2	3	4	5	6	7	8	9	10	12	11	13	14	15
Joint Strength (lb)	2130	-	2780	2900	>6380	6410	2920	2930	2350	2250	2450	3360	7350	7720	7950
Effect of Corrosion (% Change)			-4.1		>-0.5		-0.3		+4.4		-27.1		-4.8		-0.6
Failure Mode	TF	TF 9	TF	TF	NT (grip)	TF NT	TF	TF	TF NT FS	TF NT	TF 1	FS	NT	NT	NT

FAILURE DATA	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
Joint Strength (lb)	8000	7780	7510	8840	7920	7960	7890	8060	7920	5690	5850	6640	6770	7220	7600
Effect of Corrosion (% Change)		+3.6		-13.9		+0.9		+2.0		-2.7		-4.9		-5.0	
Failure Mode	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT





Specimen Number

23	25	27	29	31	33	35	37	39	41	43	45	47	49	51	53	55	57	59
- - - -	- - - -	C - - -	- - - -	- - - -	C - - -	- - - -	Bonded	Bonded	Bonded	Bonded	Bonded	Eonded	C - - -	C - - -	C - - -	C - - -	C - - -	C -
- - - -	- - - -	C - - -	- - - -	- - - -	C - - -	- - - -							C - - -	C - - -	C Y - -	C Y - -	C - - R	C -
- - - -	- - - -	C - - -	- - - -	- - - -	C - - -	- - - -							C - - -	C - - -	C Y - -	C Y - -	C - - R	C -
- - - -	- - - -	- - - -	- - - -	- - - -	- - - -	C - - -							C - - -	C - - -	C - - -	C - - -	C - - -	C -
- - - -	- - - -	- - - -	- - - -	- - - -	- - - -	C - - -							C - - -	C - - -	C Y - -	C Y - -	C - - R	C -
- - - -	- - - -	- - - -	- - - -	- - - -	- - - -	C - - -							C - - -	C - - -	C Y - -	C Y - -	C - - R	C -
- - - -	- - - -	C - - -	- - - -	- - - -	C - - -	C - - -							C - - -	C - - -	C - - -	- - - -	C - - -	C -
- - - -	- - - -	C - - -	- - - -	- - - -	C - - -	C - - -							C - - -	C - - -	C Y - -	- - - -	C - - R	C -
- - - -	- - - -	C - - -	- - - -	- - - -	C - X -	C - - -							C - - -	C - - -	C Y - -	- Y - -	C - - R	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - - -	- - - -							C - - -	C - - -	- - - -	C - - -	C - - -	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - - -	- - - -							C - - -	C - - -	- - - -	C Y - -	C - - R	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - X -	- - - -							C - - -	C - - -	- Y - -	C Y - -	C - - R	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - - -	- - - -							C - - -	C - - -	- - - -	C - - -	C - - -	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - - -	- - - -							C - - -	C - - -	- - - -	C Y - -	C - - R	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - - -	- - - -							C - - -	C - - -	- Y - -	C Y - -	C - - R	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - - -	C - - -							C - - -	C - - -	- - - -	- - - -	C - - -	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - - -	C - - -							C - - -	C - - -	- - - -	- - - -	C - - R	C -
- - - -	- - - -	- - - -	- - - -	- - - -	C - - -	C - - -							C - - -	C - - -	- Y - -	- Y - -	C - - R	C -
C - - -	C - - -	C - - -	C - - -	- - -	C - - -	C - - -							C - - -	C - - -	C - - -	C - - -	C - - -	C -
C Y - -	C Y - -	C - - -	C - - -	- - - -	C Y - -	C Y - -							C - - -	C - - -	C Y - -	C Y - -	C - - -	C -
C - - -	C Y - -	C Y - -	C Y - -	- - - -	C Y - -	C Y - -							C - - -	C - - -	C Y - -	C Y - -	C - - R	C -
C - - -	C - - -	C - - -	- - - -	- - - -	C - - -	C - - -							- - - -	- - - -	- - - -	C - - -	- - - -	- -
C - - -	C - - -	C - - -	- - - -	- - - -	C - - -	C - - -							- - - -	- - - -	- Y - -	C Y - -	- - - R	- -
C - X -	C - X -	C Y - -	- - - -	- - - -	C - - -	C - - -							- - - -	- - - -	- - - -	C - - -	- - - R	- -
- - - -	- - - -	C - - -	- - - -	- - - -	C - - -	- - - -							C - - -	C - - -	- - - -	C - - -	- - - -	C -
- - - -	- - - -	C - - -	- - - -	- - - -	C - - -	- - - -							C - - R	C - - R	- - - R	C Y - -	- - - R	C -
- - - R	- - - R	C Y - -	- - - R	- - - R	C - - R	- - - -			- - X -				C - - R	C - - R	- - - R	C - - R	- - - R	C -

Specimen Number

23	24	25	26	27	26	29	30	31	32	33	34	35	36	37	36	39	40	41
8080	7920	5690	5850	6640	6770	7220	7600	7110	6530	6100	6750	6560	5920	2200	2470	2550	2390	2400
+2.0		-2.7		-4.9		-5.0		+8.9		-9.6		+9.5		-10.9		+6.7		-5.1
NT	NT	NT	NT	NT	NT	NT	NT	NT 2	NT	NT 3	NT	NT	NT	CG	CG	CK	CK	CG

42	43	44	45	46	47	46	49	50	51	52	53	54	55	56	57	58	59
2530	3450	4900	0	0	2390	2250	6540	6300	7300	7350	6390	6350	6980	5900	6230	5900	63[illegible]
	-29.6		-		+6.2		+3.8		-0.7		+0.6		+18.3		+5.6		-[illegible]
CG	CG 4	CG	5	5	CG	CG	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT

Note: TF = Tension failure of fasteners
NT = Net graphite/epoxy section tension failure
FS = Fastener shear failure
CG = Cohesive failure in graphite/epoxy
CK = Cohesive failure in Kevlar 49
AT = Adhesive failure to titanium
CA = Cohesive failure in adhesive
1 = Anodized "spacer" adhered to graphite/epoxy causing fastener shear failure
2 = Bearing shear failure on one fastener
3 = Copper plating corroded
4 = Failure also in adhesive to aluminum
5 = Graphite/epoxy adhesive failure to copper plating
6 = Initially had net, graphite/epoxy section tension failure of grip
7 = Adhesive failure to titanium, cohesive failure in graphite/epoxy, cohesive in adhesive
8 = Adhesive failure to graphite/epoxy, cohesive failure in adhesive.
9 = Broke during cyclic testing

Butt Joint

2

Table 13. Visual Observations and Failure Loads

51	53	55	57	59	61	63	65	67	69	71	73	75	77	79	81	83	85
C - - -	C - - -	C - - -	C - - -	C - - -	C - - -	- - - -	Bonded	Bonded	Bonded	Bonded	C - - -	C - - -	- - - -	Bonded	C - - -	C - - -	Bonded
C - - -	C Y - -	C Y - -	C - - R	C - - R	C Y - -	- - - -					C Y - -	C Y - -	- - - -		C - - -	C - - R	
C - - -	C Y - -	C Y - -	C - - R	C - - R	C - - -	- - - -					C Y X -	C Y - -	- - - -		C - - -	C - - R	
C - - -	C - - -	C - - -	C - - -	C - - -	C - - -	- - - -					C - - -	C - - -	- - - -		C - - -	C - - -	
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-0.7		+0.8		+18.3		+5.8		-11.1		-2.4		+11.3		-31.7		+5.8		-11.8		+20.7		-10.1		+3.2		-11.8		+10.3		-4.8		-3.2		-22.9	
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6.3 DISCUSSION OF RESULTS

An examination of the results in Table 13 reveals that corrosion of the graphite/epoxy joint specimens was no worse than the aluminum-aluminum control specimens. The control specimens themselves had surprisingly little corrosion. For example, corrosion was noted at only two fasteners on Specimen 73 (aluminum control with aluminum fasteners, see Figures 7 and 8).

The materials and optimum processing procedure used obviously contributed to these results. Coating of the detail panels and fasteners was performed under laboratory conditions and does not reflect possible production deficiencies.

Specimens 9 and 21 show that corrosion can occur with serious results (Figures 9 and 10). These specimens had the copper/nickel plating that failed in adhesion. On Specimen 9, one of the aluminum fasteners had the head completely corroded away. There is obviously a potential for problems with these joints, but with proper protection corrosion can be controlled.

No corrosion was noted on any specimen that did not have a crack around the fastener. One of the major problems with this type of joint corrosion testing is to obtain a realistic fastener failure before corrosive environment exposure. Even though these specimens were carefully designed, assembled, and fatigue tested, cracking occurred only on a fraction of the fasteners.

Table 13 documents the observed cracks. Figure 11 shows Specimen 61 after cyclic tension loading. Cracks occurred only around some of the fasteners. In some specimens, no cracking at all was noted. Most of the specimens with no cracking were assembled using a protection system consisting of a polysulfide faying surface sealant between the two panels. As shown by the joint strength data, the sealant contributed significantly to the joint strength of Specimens 5 and 6. Sealant joints were apparently much more rigid and resistant to cracking.

A yellow residue was found on many of the specimens that had the chromate-inhibited polysulfide sealant over the fasteners (Figure 12). This was also noted at the butt joints.

An analysis of this material showed that it consisted essentially of sodium, calcium, and chromate. This was also found to be in the sealant material. No strontium was found, so apparently it is due to the sealant only. It effectively inhibited corrosion where present.

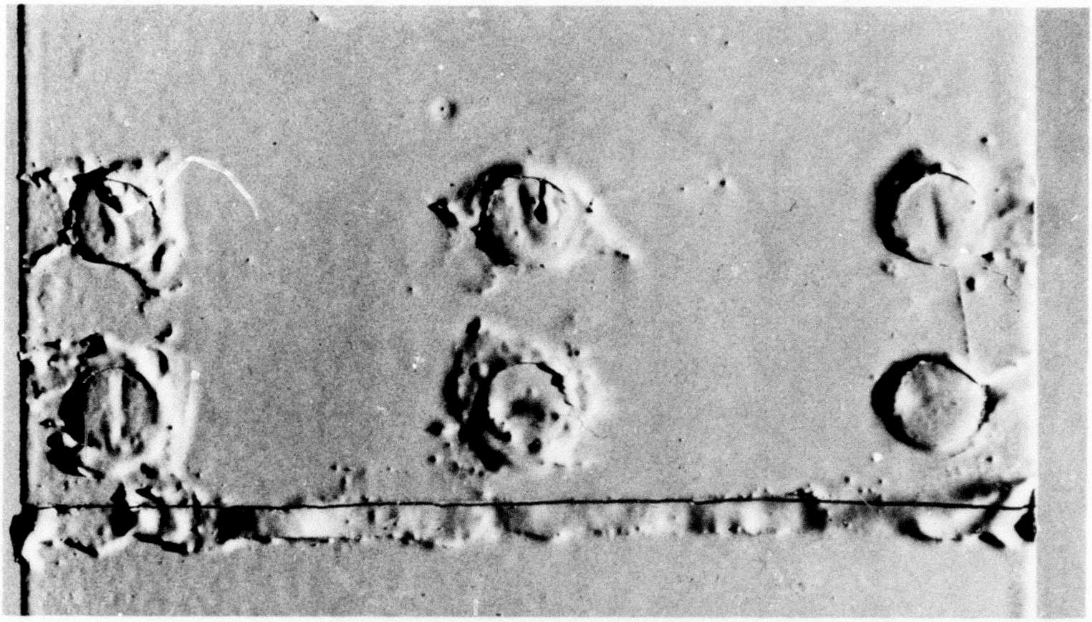

Figure 7. Specimen 73 after Cyclic Tension Loading, One Week of Humidity, and Two Weeks of Exfoliation Salt Spray (2×)

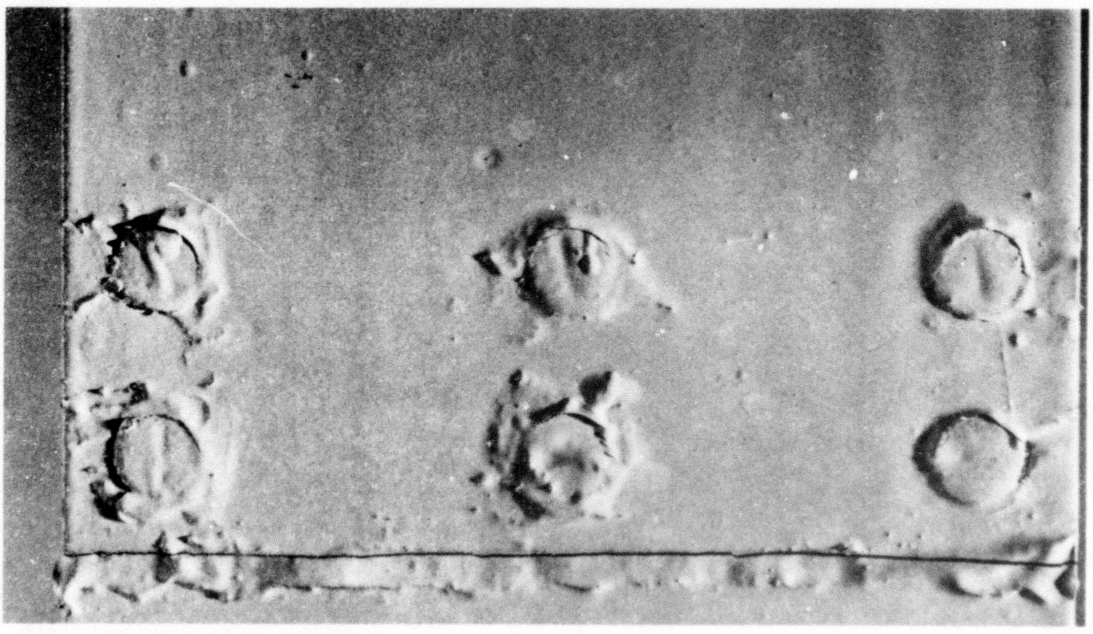

Figure 8. Specimen 73 after Cyclic Tension Loading, One Week of Humidity, and Four Weeks of Exfoliation Salt Spray (2×)

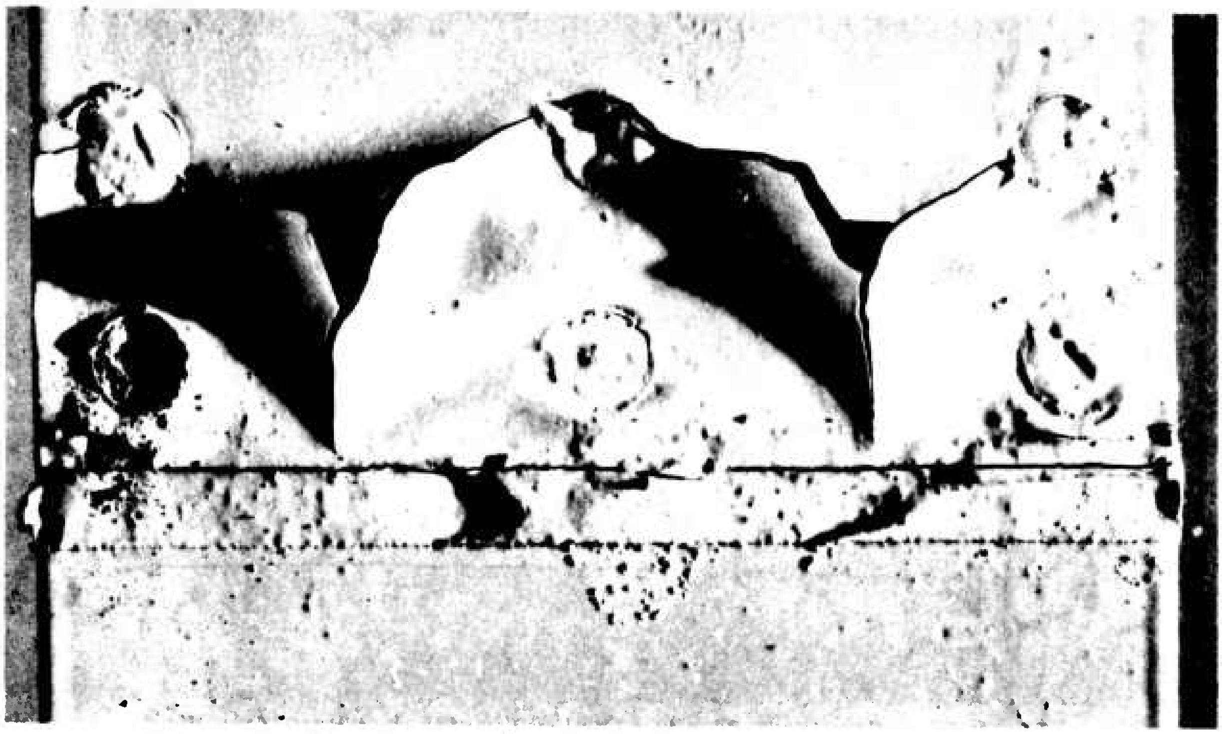

Figure 9. Specimen 9 after Cyclic Tension Loading, One Week of Humidity and Two Weeks of Exfoliation Salt Spray Showing Plating Failure and Corroded Aluminum Fastener (2x)

Figure 10. Specimen 21 after Cyclic Tension Loading, One Week of Humidity and Two Weeks of Exfoliation Salt Spray Showing Plating Failure and Corroded Cadmium Plated Steel Fastener (2x)

Figure 11. Specimen 61 after Cyclic Tension Loading Showing Partial Fastener Cracking (2 x)

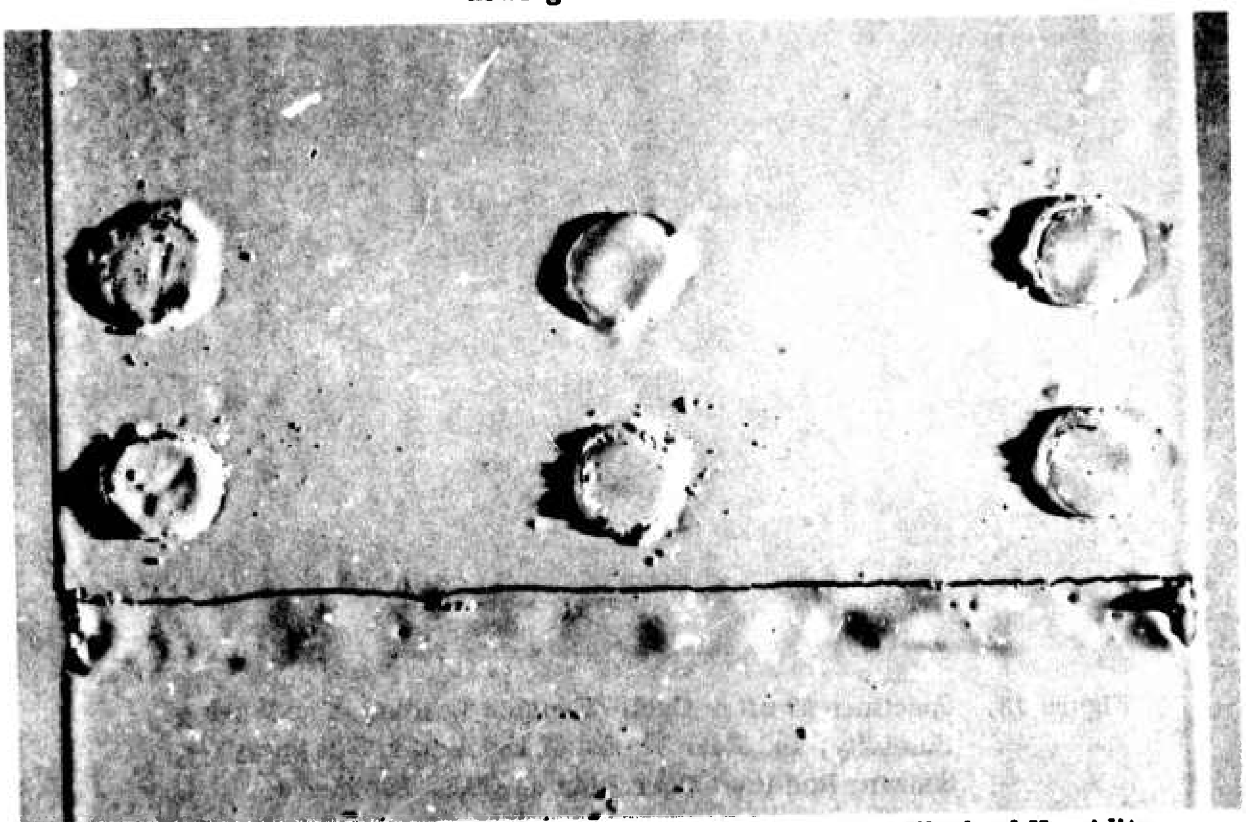

Figure 12. Specimen 3 after Cyclic Tension Loading, One Week of Humidity, and Two Weeks of Exfoliation Salt Spray Exposure Showing Sodium and Calcium Chromate Deposits Around Fasteners (2 x)

In general, the results show that corrosion was significant only in a few special cases. It appears that corrosion protection of graphite/epoxy attached to aluminum can be accomplished using conventional techniques.

The graphite/epoxy-titanium joint specimens showed no corrosion where titanium fasteners were used. However, the CRES (A286) fasteners were attacked unless protected with sealants and coatings. Even then, some red rust corrosion was noted at cracks. An examination of the CRES fasteners showed no structural attack, but they did cause extensive red rust staining of the specimen (Figure 13).

The only corrosion on an adhesive-bonded specimen is shown in Figure 14. The back side was attacked at one corner of the interface. This corrosion extended into the interface. An area 0.5 by 0.25 inch was affected. No significant loss in joint strength was noted. However, this again demonstrates the potential problem with graphite/epoxy-metal interfaces and points out the need for an adequate corrosion protection system. This specimen had only a coating for protection. It appears that more than an organic coating is necessary for reliable protection.

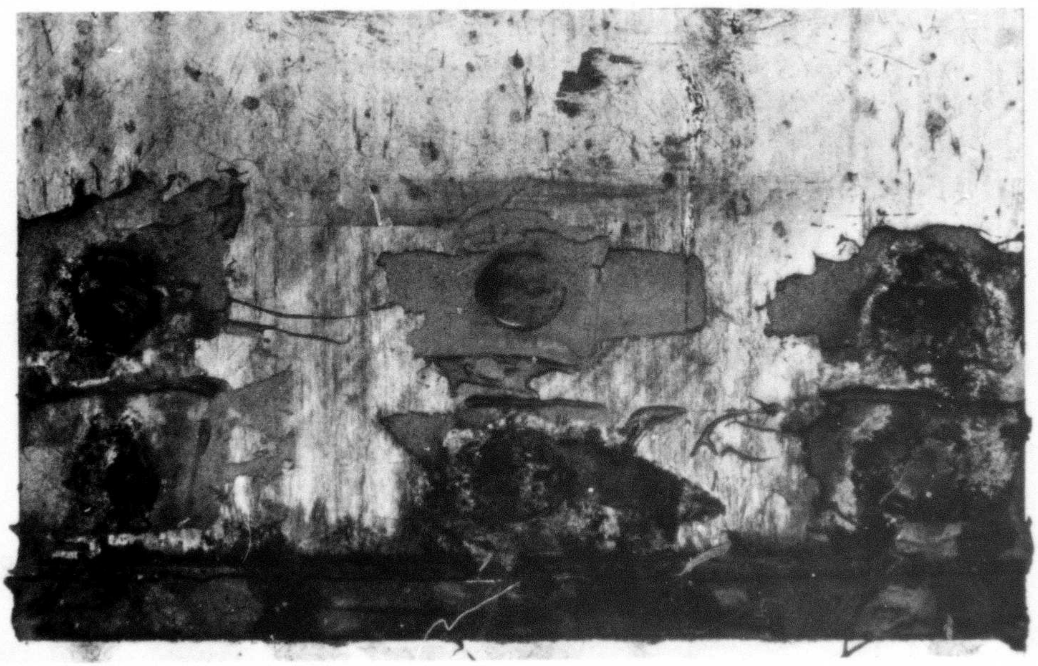

Figure 13. Specimen 57 after Cyclic Tension Loading, One Week of Humidity, and Four Weeks of Exfoliation Salt Spray Showing Red Rust Corrosion on CRES Fasteners

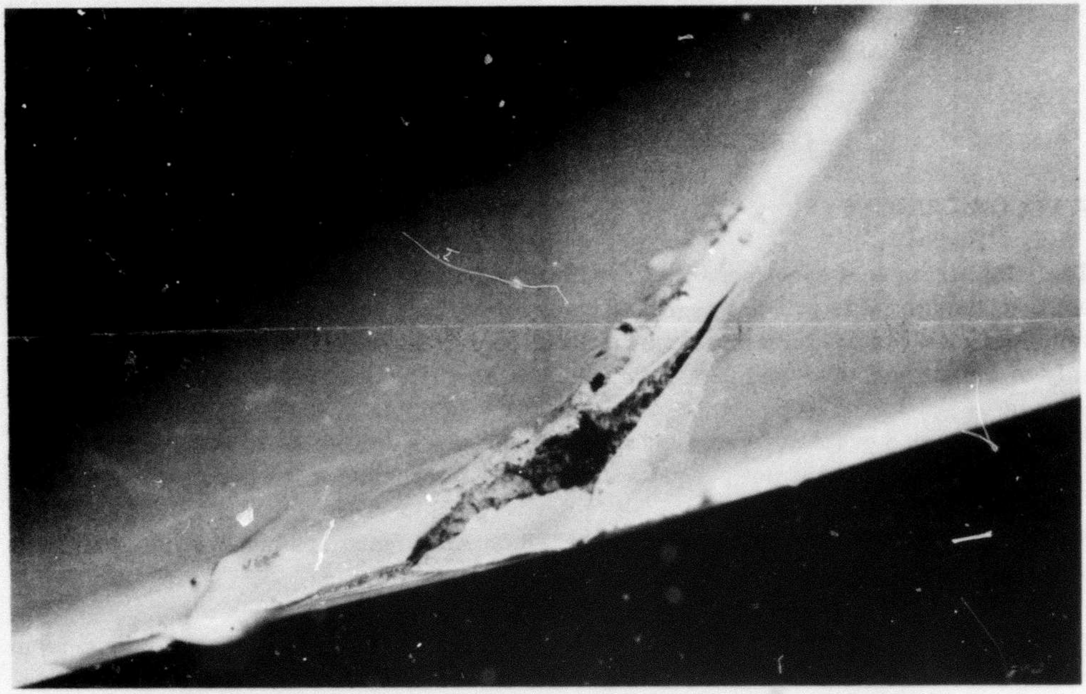

Figure 14. Specimen 41 after Cyclic Tension Loading, One Week of Humidity, and Four Weeks of Exfoliation Salt Spray Showing Corrosion on Back Side of Bond Interface (2x)

SECTION 7

CONCLUSIONS AND RECOMMENDATIONS

7.1 CONCLUSIONS

The joint strength of specimens representing graphite/epoxy-aluminum and graphite/epoxy-titanium joints was not degraded by exposure to a corrosive environment. All specimens had, as a minimum, a conventional corrosion protection system consisting only of organic coatings and sealants.

Adhesive-bonded graphite/epoxy-aluminum joints are susceptable to corrosive attack, and organic coatings are not adequate for reliable corrosion protection.

Significant red rust corrosion occurs when CRES A286 fasteners are used in graphite/epoxy-titanium joints.

Discrimination between sophisticated corrosion protection systems requires long-term exposure when environments such as humidity and exfoliation salt spray are used.

Visible corrosion at fasteners correlated well with electrical resistance measurements.

The water permeability tests and the mechanical properties tests of the finishes and sealants used in this program clearly show the advantages of polyurethane finishes for water repulsion and polysulfide sealants for elongation at -65°F.

7.2 RECOMMENDATIONS

Simple component structures should be tested under dynamic conditions of stress, temperature, and corrosive environment to verify the results of this program.

Element test panels representing the systems tested in this program should be repeated with the test time increased so that a better comparison can be made of the more sophisticated protection systems.

SECTION 8

REFERENCES

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3. S. J. Ketcham and P. W. Jeffery, "Exfoliation Corrosion Testing of 7178 and 7075 Aluminum Alloys," Localized Corrosion Cause of Metal Failure, ASTM STP 516, American Society for Testing and Materials, 1972, pp. 273-302.

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